

## WEST Search History

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DATE: Friday, June 23, 2006

<u>Hide?</u>	<u>Set Name</u>	<u>Query</u>	<u>Hit Count</u>
<i>DB=PGPB,USPT,USOC,EPAB,JPAB,DWPI; THES=ASSIGNEE; PLUR=YES; OP=ADJ</i>			
<input type="checkbox"/>	L6	l5 not l1	12
<input type="checkbox"/>	L5	L4 and tetrafluoroethylene	12
<input type="checkbox"/>	L4	L2 and ethylene	66
<input type="checkbox"/>	L3	L2 and copper with catalyst	1
<input type="checkbox"/>	L2	polyfluoroalkyl acrylate	99
<input type="checkbox"/>	L1	polyfluoroalkylethyl acrylate	2

END OF SEARCH HISTORY

## Connecting via Winsock to STN

Welcome to STN International! Enter x:x

LOGINID: ssspta1202jxp

PASSWORD :

TERMINAL (ENTER 1, 2, 3, OR ?):2

NEWS	1	Web Page URLs for STN Seminar Schedule - N. America
NEWS	2	"Ask CAS" for self-help around the clock
NEWS	3	JAN 17 Pre-1988 INPI data added to MARPAT
NEWS	4	FEB 21 STN AnaVist, Version 1.1, lets you share your STN AnaVist visualization results
NEWS	5	FEB 22 The IPC thesaurus added to additional patent databases on STN
NEWS	6	FEB 22 Updates in EPFULL; IPC 8 enhancements added
NEWS	7	FEB 27 New STN AnaVist pricing effective March 1, 2006
NEWS	8	MAR 03 Updates in PATDPA; addition of IPC 8 data without attributes
NEWS	9	MAR 22 EMBASE is now updated on a daily basis
NEWS	10	APR 03 New IPC 8 fields and IPC thesaurus added to PATDP FULL
NEWS	11	APR 03 Bibliographic data updates resume; new IPC 8 fields and IPC thesaurus added in PCTFULL
NEWS	12	APR 04 STN AnaVist \$500 visualization usage credit offered
NEWS	13	APR 12 LINSPEC, learning database for INSPEC, reloaded and enhanced
NEWS	14	APR 12 Improved structure highlighting in FQHIT and QHIT display in MARPAT
NEWS	15	APR 12 Derwent World Patents Index to be reloaded and enhanced during second quarter; strategies may be affected
NEWS	16	MAY 10 CA/CAplus enhanced with 1900-1906 U.S. patent records
NEWS	17	MAY 11 KOREAPAT updates resume
NEWS	18	MAY 19 Derwent World Patents Index to be reloaded and enhanced
NEWS	19	MAY 30 IPC 8 Rolled-up Core codes added to CA/CAplus and USPAT FULL/USPAT2
NEWS	20	MAY 30 The F-Term thesaurus is now available in CA/CAplus
NEWS	21	JUN 02 The first reclassification of IPC codes now complete in INPADOC
NEWS EXPRESS		FEBRUARY 15 CURRENT VERSION FOR WINDOWS IS V8.01a, CURRENT MACINTOSH VERSION IS V6.0c(ENG) AND V6.0Jc(JP), AND CURRENT DISCOVER FILE IS DATED 19 DECEMBER 2005. V8.0 AND V8.01 USERS CAN OBTAIN THE UPGRADE TO V8.01a AT <a href="http://download.cas.org/express/v8.0-Discover/">http://download.cas.org/express/v8.0-Discover/</a>
NEWS HOURS		STN Operating Hours Plus Help Desk Availability
NEWS LOGIN		Welcome Banner and News Items
NEWS IPC8		For general information regarding STN implementation of IPC 8
NEWS X25		X.25 communication option no longer available after June 2006

Enter NEWS followed by the item number or name to see news on that specific topic.

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FILE 'HOME' ENTERED AT 17:54:16 ON 23 JUN 2006

=> file casreact  
COST IN U.S. DOLLARS  
  
FULL ESTIMATED COST

SINCE FILE ENTRY	TOTAL SESSION
0.21	0.21

FILE 'CASREACT' ENTERED AT 17:54:27 ON 23 JUN 2006  
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FILE CONTENT:1840 - 18 Jun 2006 VOL 144 ISS 25

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\*\*\*\*\*  
\*  
\* CASREACT now has more than 10 million reactions  
\*  
\*\*\*\*\*

Some CASREACT records are derived from the ZIC/VINITI database (1974-1991) provided by InfoChem, INPI data prior to 1986, and Biotransformations database compiled under the direction of Professor Dr. Klaus Kieslich.

This file contains CAS Registry Numbers for easy and accurate substance identification.

```
=> Uploading C:\Program Files\Stnexp\Queries\10516940a.str
```

## L1 STRUCTURE UPLOADED

```
=> d 11
L1 HAS NO ANSWERS
L1           STR
*** STRUCTURE DIAGRAM IS NOT AVAILABLE ***
```

Structure attributes must be viewed using STN Express query preparation.

=> s 11  
SAMPLE SEARCH INITIATED 17:55:04 FILE 'CASREACT'  
SCREENING COMPLETE - 246 REACTIONS TO VERIFY FROM 25 DOCUMENTS

100.0% DONE 246 VERIFIED 0 HIT RXNS . 0 DOCS  
SEARCH TIME: 00.00.01

FULL FILE PROJECTIONS: ONLINE \*\*COMPLETE\*\*  
                          BATCH \*\*COMPLETE\*\*  
PROJECTED VERIFICATIONS: 3980 TO 5860  
PROJECTED ANSWERS: 0 TO 0

=> s 11 full  
FULL SEARCH INITIATED 17:55:17 FILE 'CASREACT'

## Connecting via Winsock to STN

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\* \* \* \* \* \* \* \* \* \* \* \* \* \* \* \* STN Columbus \* \* \* \* \* \* \* \* \* \* \* \* \* \* \*

FILE 'HOME' ENTERED AT 10:32:28 ON 23 JUN 2006

=> file caplus	SINCE FILE	TOTAL
COST IN U.S. DOLLARS	ENTRY	SESSION
FULL ESTIMATED COST	0.21	0.21

FILE 'CAPLUS' ENTERED AT 10:32:46 ON 23 JUN 2006  
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FILE COVERS 1907 - 23 Jun 2006 VOL 145 ISS 1  
FILE LAST UPDATED: 22 Jun 2006 (20060622/ED)

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=> polyfluoroalkylethyl iodide  
POLYFLUOROALKYLETHYL IS NOT A RECOGNIZED COMMAND  
The previous command name entered was not recognized by the system.  
For a list of commands available to you in the current file, enter  
"HELP COMMANDS" at an arrow prompt (>).

=> s polyfluoroalkylethyl iodide  
1 POLYFLUOROALKYLETHYL  
173365 IODIDE  
24137 IODIDES  
183154 IODIDE  
(IODIDE OR IODIDES)  
L1 1 POLYFLUOROALKYLETHYL IODIDE  
(POLYFLUOROALKYLETHYL (W) IODIDE)

=> d 11 ibib ab

L1 ANSWER 1 OF 1 CAPLUS COPYRIGHT 2006 ACS on STN  
ACCESSION NUMBER: 2003:1006848 CAPLUS  
DOCUMENT NUMBER: 140:17105  
TITLE: Metallic copper catalyst for  
polyfluoroalkylethyl iodide  
production and process for producing  
polyfluoroalkylethyl iodides  
INVENTOR(S): Funakoshi, Yoshiro; Miki, Jun  
PATENT ASSIGNEE(S): Daikin Industries, Ltd., Japan  
SOURCE: PCT Int. Appl., 21 pp.  
CODEN: PIXXD2  
DOCUMENT TYPE: Patent  
LANGUAGE: Japanese  
FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
WO 2003106023	A1	20031224	WO 2003-JP7643	20030617
W: AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NI, NO, NZ, OM, PG, PH, PL, PT, RO, RU, SC, SD, SE, SG, SK, SL, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, YU, ZA, ZM, ZW				
RW: GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZM, ZW, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM, AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HU, IE, IT, LU, MC, NL, PT, RO, SE, SI, SK, TR, BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG				
AU 2003241701	A1	20031231	AU 2003-241701	20030617
CN 1662302	A	20050831	CN 2003-814144	20030617
US 2005250966	A1	20051110	US 2004-516940	20041215
PRIORITY APPLN. INFO.:			JP 2002-175381	A 20020617
			WO 2003-JP7643	W 20030617

OTHER SOURCE(S): MARPAT 140:17105

AB Copper catalyzes the addition of ethylene to polyfluoroalkyl iodides to prepare polyfluoroalkylethyl iodides. Thus, 138.6 g C2F5I, 8.21 g Cu, and 1.0 MPa C2H4 were heated 80 min at 80° to give perfluoroethylethyl iodide at selectivity 99.8% and C2F5I conversion 99.5%.

REFERENCE COUNT: 32 THERE ARE 32 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

=> s polyfluoroalkyl iodide  
 1572 POLYFLUOROALKYL  
 1 POLYFLUOROALKYLS  
 1572 POLYFLUOROALKYL  
 (POLYFLUOROALKYL OR POLYFLUOROALKYLS)  
 173365 IODIDE  
 24137 IODIDES  
 183154 IODIDE  
 (IODIDE OR IODIDES)  
 L2 89 POLYFLUOROALKYL IODIDE  
 (POLYFLUOROALKYL (W) IODIDE)

=> s l2 and ethylene  
 524665 ETHYLENE  
 3356 ETHYLENES  
 526141 ETHYLENE  
 (ETHYLENE OR ETHYLENES)  
 L3 5 L2 AND ETHYLENE

=> s l3 ibib ab 1-5  
 MISSING OPERATOR L3 IBIB  
 The search profile that was entered contains terms or nested terms that are not separated by a logical operator.

=> d l3 ibib ab 1-5

L3 ANSWER 1 OF 5 CAPLUS COPYRIGHT 2006 ACS on STN  
 ACCESSION NUMBER: 2003:1006848 CAPLUS  
 DOCUMENT NUMBER: 140:17105  
 TITLE: Metallic copper catalyst for polyfluoroalkylethyl iodide production and process for producing polyfluoroalkylethyl iodides  
 INVENTOR(S): Funakoshi, Yoshiro; Miki, Jun  
 PATENT ASSIGNEE(S): Daikin Industries, Ltd., Japan

SOURCE: PCT Int. Appl., 21 pp.

CODEN: PIXXD2

DOCUMENT TYPE: Patent

LANGUAGE: Japanese

FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
WO 2003106023	A1	20031224	WO 2003-JP7643	20030617
W: AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NI, NO, NZ, OM, PG, PH, PL, PT, RO, RU, SC, SD, SE, SG, SK, SL, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, YU, ZA, ZM, ZW				
RW: GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZM, ZW, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM, AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HU, IE, IT, LU, MC, NL, PT, RO, SE, SI, SK, TR, BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG				
AU 2003241701	A1	20031231	AU 2003-241701	20030617
CN 1662302	A	20050831	CN 2003-814144	20030617
US 2005250966	A1	20051110	US 2004-516940	20041215
PRIORITY APPLN. INFO.:			JP 2002-175381	A 20020617
			WO 2003-JP7643	W 20030617

OTHER SOURCE(S): MARPAT 140:17105

AB Copper catalyzes the addition of ethylene to polyfluoroalkyl iodides to prepare polyfluoroalkylethyl iodides. Thus, 138.6 g C2F5I, 8.21 g Cu, and 1.0 MPa C2H4 were heated 80 min at 80° to give perfluoroethyl iodide at selectivity 99.8% and C2F5I conversion 99.5%.

REFERENCE COUNT: 32 THERE ARE 32 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L3 ANSWER 2 OF 5 CAPLUS COPYRIGHT 2006 ACS on STN

ACCESSION NUMBER: 1974:571361 CAPLUS

DOCUMENT NUMBER: 81:171361

TITLE: N-(Fluoroalkyl)arylamine dye intermediates

INVENTOR(S): Sureau, Robert; Pechmeze, Jacques

PATENT ASSIGNEE(S): Ugine Kuhlmann

SOURCE: Ger. Offen., 15 pp.

CODEN: GWXXBX

DOCUMENT TYPE: Patent

LANGUAGE: German

FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
DE 2353293	A1	19740516	DE 1973-2353293	19731024
FR 2205894	A5	19740531	FR 1972-38896	19721103
CH 590204	A	19770729	CH 1973-14842	19731022
BE 806852	A1	19740430	BE 1973-137359	19731031
NL 7314986	A	19740507	NL 1973-14986	19731101
JP 49108028	A2	19741014	JP 1973-123920	19731102
GB 1448701	A	19760908	GB 1973-50911	19731102
IT 999712	A	19760310	IT 1973-70216	19731105

PRIORITY APPLN. INFO.:

AB Twelve RR1N(CH<sub>2</sub>)<sub>2</sub>(CF<sub>2</sub>)<sub>n</sub>CF<sub>3</sub> (I; R = Ph, 3-MeC<sub>6</sub>H<sub>4</sub>, 3-ClC<sub>6</sub>H<sub>4</sub>, or 1-C<sub>10</sub>H<sub>7</sub>; R<sub>1</sub> = H, Et, CH<sub>2</sub>CH<sub>2</sub>CN, or CH<sub>2</sub>CH<sub>2</sub>OH; n = 1, 3, or 5) were prepared. Thus, reaction of PhNH<sub>2</sub> with CF<sub>3</sub>CF<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>I gave 80.7% N-(3,3,4,4,4-pentafluorobutyl)aniline (II) [52671-65-5]. Similarly prepared were 4 other I. Ethylation of II gave 84.7% N-ethyl-N-(3,3,4,4,4-pentafluorobutyl)aniline [52298-66-5]. Similarly prepared was 1 other I.

Reaction of II with CH<sub>2</sub>:CHCN gave 70.2% N-(cyanoethyl)-N-(3,3,4,4,4-pentafluorobutyl)aniline [52298-65-4]. Similarly prepared were 2 other I. Reaction of N-(3,3,4,4,4-pentafluorobutyl)-m-toluidine with ethylene oxide gave 75.5% N-(pentafluorobutyl)-N-(hydroxyethyl)-m-toluidine [52671-66-6]. Similarly prepared was 1 other I.

L3 ANSWER 3 OF 5 CAPLUS COPYRIGHT 2006 ACS on STN

ACCESSION NUMBER: 1974:519901 CAPLUS  
 DOCUMENT NUMBER: 81:119901  
 TITLE: Polyfluoroalkyl iodides  
 INVENTOR(S): Jaeger, Horst  
 PATENT ASSIGNEE(S): Ciba-Geigy A.-G.  
 SOURCE: Ger. Offen., 36 pp.  
 CODEN: GWXXBX  
 DOCUMENT TYPE: Patent  
 LANGUAGE: German  
 FAMILY ACC. NUM. COUNT: 1  
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
DE 2361807	A1	19740627	DE 1973-2361807	19731212
CH 576922	A	19760630	CH 1972-18662	19721221
CA 1023768	A1	19780103	CA 1973-187808	19731210
US 3979469	A	19760907	US 1973-425011	19731214
GB 1411200	A	19751022	GB 1973-58328	19731217
FR 2211430	A1	19740719	FR 1973-45565	19731219
BE 808878	A1	19740620	BE 1973-139076	19731220
SU 528029	D	19760905	SU 1973-1978254	19731220
JP 49088811	A2	19740824	JP 1973-142552	19731221
IT 1000773	A	19760410	IT 1973-54542	19731221
PRIORITY APPLN. INFO.:			CH 1972-18662	A 19721221
			CH 1973-2653	A 19730223
			CH 1973-16037	A 19731114

AB Twenty-four F<sub>3</sub>C(CF<sub>2</sub>)<sub>n</sub>(CH<sub>2</sub>CR<sub>1</sub>F)<sub>m</sub>CH<sub>2</sub>CHR<sub>1</sub> (n = 3-11, m = 1-3, R = H, Me, or CH<sub>2</sub>OH; R<sub>1</sub> = H or F) were prepared by addition reaction of CH<sub>2</sub>:CHR to F<sub>3</sub>C(CF<sub>2</sub>)<sub>n</sub>(CH<sub>2</sub>CR<sub>1</sub>F)<sub>m</sub>I at 100-60° and 1-30 kg/cm<sup>2</sup> in the presence of Al<sub>2</sub>O<sub>3</sub>-supported catalysts containing CuCl<sub>x</sub>, (di)ethanolamine, and optionally SnCl<sub>4</sub>. Thus, reaction of F<sub>3</sub>C(CF<sub>2</sub>)<sub>3</sub>CH<sub>2</sub>CF<sub>2</sub>I with CH<sub>2</sub>:CH<sub>2</sub> over a CuCl<sub>x</sub>-SnCl<sub>4</sub>-HOCH<sub>2</sub>CH<sub>2</sub>NH<sub>2</sub> catalyst 7 hr at 140° and 26 kg/cm<sup>2</sup> gave 98.5% F<sub>3</sub>C(CF<sub>2</sub>)<sub>3</sub>CH<sub>2</sub>CF<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>I.

L3 ANSWER 4 OF 5 CAPLUS COPYRIGHT 2006 ACS on STN

ACCESSION NUMBER: 1971:22415 CAPLUS  
 DOCUMENT NUMBER: 74:22415  
 TITLE: Thermal addition of polyfluoroalkyl iodides to ethylene  
 INVENTOR(S): Bloechl, Walter  
 PATENT ASSIGNEE(S): FMC Corp.  
 SOURCE: U.S., 3 pp.  
 CODEN: USXXAM  
 DOCUMENT TYPE: Patent  
 LANGUAGE: English  
 FAMILY ACC. NUM. COUNT: 1  
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
US 3535393	A	19701020	US 1968-719692	19680408
PRIORITY APPLN. INFO.:			US 1968-719692	A 19680408

AB CH<sub>2</sub>:CH<sub>2</sub> (14 g) and 87 g n-C<sub>7</sub>F<sub>15</sub>I at 124 ml/min and atmospheric pressure were passed through a Monel spiral preheater at 180-200° and through a Pyrex reactor 60 + 1.5 cm. heated to 379-401° with a spirally-wound Nichrome wire (resistance 2.4 ohms/ft.), residence time 17

sec. The exit gas was condensed to give 91 g n-C<sub>7</sub>F<sub>15</sub>CH<sub>2</sub>CH<sub>2</sub>I, b<sub>20</sub> 89-90°, purity 90%. Similarly was prepared 87% n-C<sub>8</sub>F<sub>17</sub>CH<sub>2</sub>CH<sub>2</sub>I, m. 50°, b<sub>10</sub> 90-1°.

L3 ANSWER 5 OF 5 CAPLUS COPYRIGHT 2006 ACS on STN  
ACCESSION NUMBER: 1964:410993 CAPLUS  
DOCUMENT NUMBER: 61:10993  
ORIGINAL REFERENCE NO.: 61:1755d-h,1756a-b  
TITLE: Improvement in the preparation of perfluoroalkyl iodides from tetrafluoroethylene  
INVENTOR(S): Parsons, Raymond E.  
PATENT ASSIGNEE(S): E. I. du Pont de Nemours & Co.  
SOURCE: 5 pp.  
DOCUMENT TYPE: Patent  
LANGUAGE: Unavailable  
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
US 3132185	-----	19640505	US 1962-191722	19620502
BE 640970	-----		BE	
GB 998235	-----		GB	

AB The preparation is given of perfluoroethyl iodide (C<sub>2</sub>F<sub>5</sub>I) (I) and longer chain title compds. C<sub>2</sub>F<sub>5</sub>(CF<sub>2</sub>CF<sub>2</sub>)<sub>n</sub>I (II), where n = 1 or greater, according to the reaction 2I<sub>2</sub> + IF<sub>5</sub> + 5CF<sub>2</sub>:CF<sub>2</sub> → 5CF<sub>3</sub>CF<sub>2</sub>I. If the molar ratio of iodine to IF<sub>5</sub> <2:1, products of formula II (mixture, various n values) result, usually in conjunction with varying amts. of I depending on the iodine to IF<sub>5</sub> ratio. Thus, 0.66 mole iodine, 0.33 mole IF<sub>5</sub> (III), and 0.0028 mole SbF<sub>5</sub> were mixed at ambient temperature in a Hastelloy-C lined autoclave (2 moles iodine and 8.48 millimoles catalyst/mole III), the whole cooled <0°, evacuated to remove air, heated to 80° with agitation and CF<sub>2</sub>CF<sub>2</sub> (IV) and 192 parts added slowly; the pressure rose to 200-50 lb./in.<sup>2</sup> gage in <3 min., the temperature rose 8-26° after each addition in about 1 min. and then reverted to about 80° until 165 parts had been added. No temperature increase occurred during the addition of

the

remaining 27 parts of IV. The mixture was kept 1 hr. at 80°, cooled and the volatile products collected in a refrigerated receiver at -60°. Mass-spectrometry, showed 98.3% I, 0.4% unreacted IV, 0.8% IF<sub>2</sub>CCF<sub>2</sub>I (V) and 0.4% perfluorocyclobutane (VI). The noncondensed gases contained 28.1 parts IV and 4.3 parts I. The yield of I was 98.5%, based on IV. When the reaction temperature was 60° rather than 80°, the condensed product contained 97.4% I, 0.7% IV, 0.6% VI, 0.7% V, and 0.4% other products; the yield of I was 98.1%. When the reaction was repeated with omission of the SbF<sub>5</sub>, the condensed product contained 98.5% I, 0.4% IV, 0.5% V and 0.5% VI, the oily residue contained 94.6% V and 4.4% I and the yield of I was 98.4% based on IV. The reaction was repeated with 0.0128 mole anhydrous SnF<sub>2</sub>; the condensed product contained 96.9% I, 0.6% IV, 0.8% VI, 0.3% V, and 1.3% other products. The yield of I was 99% based on IV. Similarly, other catalysts were used (catalyst, and amount (parts) and % yield of I given): CaF<sub>2</sub>, 2.0, 78; CrF<sub>3</sub>.3H<sub>2</sub>O, 2.0, 16; KF, 2.0, 18; HF, 100.0, 15; CoF<sub>2</sub>.2H<sub>2</sub>O, 2.0, 60; PbF<sub>4</sub>, 2.0, 39; CuF<sub>2</sub>, 2.0, 78; NiF<sub>2</sub>.5H<sub>2</sub>O, 2.0, 28; and AgF (50%), 2.0, 73. The following examples give the mixture of components. A mixture of 0.33 mole iodine, 0.182 mole III, and 0.0028 mole SbF<sub>3</sub> (VII) was prepared at ambient temperature in a Hastelloy-C lined autoclave,

cooled to below 0°, evacuated, sealed, heated to 60° with agitation, and 1.21 moles IV added slowly in small increments in 1 hr. The reaction was exothermic, the pressure dropped rapidly from 150-250 lb./in.<sup>2</sup> gage to <100 lb./in.<sup>2</sup> gage, the temperature rose 7-44° after each addition. The mixture was kept at 70° 1 hr., cooled, the volatile products were vented through a H<sub>2</sub>O scrubber and a CaSO<sub>4</sub> drying column, condensed in a refrigerated receiver at -60° and the condensed volatile material analyzed. It contained (in mole-%) 95.1 I, 1.2 C<sub>4</sub>F<sub>9</sub>I

(VIII), 0.5 IV, and 1 C2F6 (IX). The noncondensed gases were found to consist of 8.0 parts IV, 4.0 parts I, and 3.1 parts IX. The nonvolatile residue (100 parts) was washed with H<sub>2</sub>O and analyzed with a vapor phase chromatograph (in weight-%): I 21.1; VIII 28.7; C<sub>6</sub>F<sub>13</sub>I (X) 20.6; C<sub>8</sub>F<sub>17</sub>I (XI) 13.0; C<sub>10</sub>F<sub>21</sub>I (XII) 7.5; C<sub>12</sub>F<sub>25</sub>I (XIII) 4.1; C<sub>14</sub>F<sub>29</sub>I (XIV) 2.3; C<sub>16</sub>F<sub>33</sub>I (XV) 1.1. The combination of these analyses indicates that 96.9% IV is accounted for; % yields are as follows (based on IV consumed): I 48.8, VIII 15.8, X 12.3, XI 1.7, XV 0.9, and IX 0.6. Thus, the total yield of I was 95.8% and the total yield of products II was 47.1%. Iodine consumption was quant. Similarly, addnl. expts. were carried out with a varying ratio of iodine and III, as well as the temperature and the catalysts.

=> d his

(FILE 'HOME' ENTERED AT 10:32:28 ON 23 JUN 2006)

FILE 'CAPLUS' ENTERED AT 10:32:46 ON 23 JUN 2006

L1 1 S POLYFLUOROALKYLETHYL IODIDE  
L2 89 S POLYFLUOROALKYL IODIDE  
L3 5 S L2 AND ETHYLENE

=> s tetrafluoroethylene and 12

25843 TETRAFLUOROETHYLENE  
34 TETRAFLUOROETHYLENES  
25850 TETRAFLUOROETHYLENE  
(TETRAFLUOROETHYLENE OR TETRAFLUOROETHYLENES)

L4 8 TETRAFLUOROETHYLENE AND L2

=> s 14 not 13

L5 6 L4 NOT L3

=> d 15 ibib ab 1-6

L5 ANSWER 1 OF 6 CAPLUS COPYRIGHT 2006 ACS on STN  
ACCESSION NUMBER: 1997:381429 CAPLUS  
DOCUMENT NUMBER: 127:81136  
TITLE: Pentafluoro- $\lambda^6$ -sulfanyl (SF<sub>5</sub>) fluoroalkyl iodides  
AUTHOR(S): Terjeson, Robin J.; Renn, Julia; Winter, Rolf; Gard, Gary L.  
CORPORATE SOURCE: Dep. Chem., Portland State Univ., Portland, OR, 97207, USA  
SOURCE: Journal of Fluorine Chemistry (1997), 82(1), 73-78  
CODEN: JFLCAR; ISSN: 0022-1139  
PUBLISHER: Elsevier  
DOCUMENT TYPE: Journal  
LANGUAGE: English  
OTHER SOURCE(S): CASREACT 127:81136

AB The synthesis of several SF<sub>5</sub>-containing fluoroalkyl iodides derived from an improved synthesis of SF<sub>5</sub>CF<sub>2</sub>CF<sub>2</sub>I are reported. These include SF<sub>5</sub>CF<sub>2</sub>CF<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>I, SF<sub>5</sub>(CF<sub>2</sub>)<sub>4</sub>I, SF<sub>5</sub>CF<sub>2</sub>CF<sub>2</sub>(CH<sub>2</sub>)<sub>4</sub>I, SF<sub>5</sub>CF<sub>2</sub>CF<sub>2</sub>CH:CHI and SF<sub>5</sub>CF<sub>2</sub>CF<sub>2</sub>CHFCF<sub>2</sub>I. The higher homologs SF<sub>5</sub>(CF<sub>2</sub>)<sub>6</sub>I, SF<sub>5</sub>CF<sub>2</sub>CF<sub>2</sub>(CHFCF<sub>2</sub>)<sub>2</sub>I and SF<sub>5</sub>CF<sub>2</sub>CF<sub>2</sub>(CH<sub>2</sub>CH<sub>2</sub>)<sub>3</sub>I were also identified.

REFERENCE COUNT: 18 THERE ARE 18 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L5 ANSWER 2 OF 6 CAPLUS COPYRIGHT 2006 ACS on STN  
ACCESSION NUMBER: 1996:137648 CAPLUS  
DOCUMENT NUMBER: 124:177222  
TITLE: Initiators for telomerization of polyfluoroalkyl iodides with fluoroolefins  
INVENTOR(S): Krespan, Carl George; Petrov, Viacheslav Alexandrovic;

PATENT ASSIGNEE(S): Smart, Bruce Edmund  
 du Pont de Nemours, E. I., and Co., USA  
 SOURCE: PCT Int. Appl., 14 pp.  
 CODEN: PIXXD2  
 DOCUMENT TYPE: Patent  
 LANGUAGE: English  
 FAMILY ACC. NUM. COUNT: 1  
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
WO 9532936	A1	19951207	WO 1995-US5857	19950518
W: AU, CA, CN, JP, KR				
RW: AT, BE, CH, DE, DK, ES, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE				
US 5574193	A	19961112	US 1995-416942	19950404
AU 9525857	A1	19951221	AU 1995-25857	19950518
PRIORITY APPLN. INFO.:			US 1994-249311	A 19940526
			WO 1995-US5857	W 19950518

AB Polyfluoroalkyl iodides are telomerized with polyfluoroolefins in the presence of strong fluorooxidizer initiators. The products obtained are useful in the production of surfactants and oil repellents. Tetrafluoroethylene and perfluorobutyl iodide were telomerized in the presence of  $XeF_2$ .

L5 ANSWER 3 OF 6 CAPLUS COPYRIGHT 2006 ACS on STN  
 ACCESSION NUMBER: 1988:21312 CAPLUS  
 DOCUMENT NUMBER: 108:21312  
 TITLE: Studies of fluoroalkylation and fluoroalkoxylation. Part 16. Reactions of fluoroalkyl iodides with some nucleophiles by SRN1 mechanism  
 AUTHOR(S): Chen, Qing Yun; Qiu, Zai Ming  
 CORPORATE SOURCE: Shanghai Inst. Org. Chem., Acad. Sin., Shanghai, Peop. Rep. China  
 SOURCE: Journal of Fluorine Chemistry (1987), 35(2), 343-57  
 CODEN: JFLCAR; ISSN: 0022-1139  
 DOCUMENT TYPE: Journal  
 LANGUAGE: English  
 OTHER SOURCE(S): CASREACT 108:21312

AB  $XCF_2CF_2I$  [I;  $X = ClCF_2CF_2, Cl(CF_2)_4$ ] readily react with the anion of Et acetoacetate in DMF to give  $XCF_2C(CH_2CO_2Et) : CHCO_2Et$  (II),  $XCF_2CF_2H$  (III),  $Ac_2CHCO_2Et$ , and  $AcCH(CO_2Et)_2CH(CO_2Et)Ac$  (IV). The reaction can be partly suppressed with 1,4-dinitrobenzene and the radical intermediate can be trapped by diallyl ether (DAE) to give the THF derivs. Anions of acetylacetone and malonitrile react also with I in the presence of DAE to yield the five-membered ring compds. All these results seem to indicate that the reaction is a radical chain process induced by electron transfer. In the case of  $XCF_2CF_2I$  (I;  $X = Cl$ ), tetrafluoroethylene and IV, instead of II and III, are produced, showing the occurrence of  $\beta$ -fragmentation of the 2-halotetrafluoroethyl radical in the initiation step.

L5 ANSWER 4 OF 6 CAPLUS COPYRIGHT 2006 ACS on STN  
 ACCESSION NUMBER: 1977:422253 CAPLUS  
 DOCUMENT NUMBER: 87:22253  
 TITLE: Methyl-terminated perfluoroalkyl iodides and related compounds  
 AUTHOR(S): Rondestvedt, Christian S., Jr.  
 CORPORATE SOURCE: Org. Chem. Dep., E. I. du Pont de Nemours and Co., Wilmington, DE, USA  
 SOURCE: Journal of Organic Chemistry (1977), 42(11), 1985-90  
 CODEN: JOCEAH; ISSN: 0022-3263  
 DOCUMENT TYPE: Journal  
 LANGUAGE: English  
 AB 1,1-Difluoroethyl iodide was prepared from vinylidene fluoride and hydrogen

iodide in 96% yield; it telomerized with tetrafluoroethylene with acyl peroxide catalysis to give the resp. Me(CF<sub>2</sub>)<sub>n</sub>I (n = 3,5,7,9,11,13,15).

L5 ANSWER 5 OF 6 CAPLUS COPYRIGHT 2006 ACS on STN  
ACCESSION NUMBER: 1970:476644 CAPLUS  
DOCUMENT NUMBER: 73:76644  
TITLE: Linear polyfluoroalkyl iodide compounds  
INVENTOR(S): Rebsdat, Siegfried; Schuierer, Erich; Hahn, Helmut  
PATENT ASSIGNEE(S): Farbwerte Hoechst A.-G.  
SOURCE: Ger., 4 pp.  
CODEN: GWXXAW  
DOCUMENT TYPE: Patent  
LANGUAGE: German  
FAMILY ACC. NUM. COUNT: 1  
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
DE 1915395	A	19700702	DE 1969-1915395	19690326
NL 7004025	A	19700929	NL 1970-4025	19700320
BE 748010	A	19700928	BE 1970-748010	19700326
FR 2035913	A5	19701224	FR 1970-10881	19700326
FR 2035913	B1	19740503		
GB 1256818	A	19711215	GB 1970-1256818	19700326

PRIORITY APPLN. INFO.: DE 1969-1915395 A 19690326  
AB Compds. of the general formula, C<sub>2</sub>F<sub>5</sub>(CF<sub>2</sub>CF<sub>2</sub>)<sub>n</sub>I, where n = 2 to 5, were prepared by treating C<sub>2</sub>F<sub>5</sub>I with tetrafluoroethylene in the presence of 0.08-0.2% bis(trichloroacryloyl) peroxide, at 60-75° and 8-12 atmospheric. The products are intermediates for the production of hydro- and oleophobic media.

L5 ANSWER 6 OF 6 CAPLUS COPYRIGHT 2006 ACS on STN  
ACCESSION NUMBER: 1969:11105 CAPLUS  
DOCUMENT NUMBER: 70:11105  
TITLE: Polyfluoroalkyl iodides from tetrafluoroethylene and lower polyfluoroalkyl iodides  
INVENTOR(S): Blochl, Walter  
PATENT ASSIGNEE(S): FMC Corp.  
SOURCE: U.S., 5 pp.  
CODEN: USXXAM  
DOCUMENT TYPE: Patent  
LANGUAGE: English  
FAMILY ACC. NUM. COUNT: 1  
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
US 3404189	A	19681001	US 1966-603072	19661104

PRIORITY APPLN. INFO.: US 1966-603072 A 19661104  
AB A 3:1 molar mixture of 29.1 g. n-C<sub>7</sub>H<sub>15</sub>I (I) and Fe<sub>2</sub>C:CF<sub>2</sub> were heated to 200° and passed through a glass tube 1.5 m. + 6 mm. at 355° and 80 mm., residence time 12 min., to give a condensate of 26.65 g. I, 3.64 g. n-C<sub>9</sub>F<sub>19</sub>I, and 0.53 g. 90% n-C<sub>11</sub>F<sub>23</sub>I. Similarly were prepared 13% H(CF<sub>2</sub>)<sub>4</sub>I, b<sub>750</sub> 85°; 86% Cl(CF<sub>2</sub>)<sub>4</sub>I, b<sub>350</sub> 70°; 2.7 g. I(CF<sub>2</sub>)<sub>4</sub>I, from 20 g. ICF<sub>2</sub>CF<sub>2</sub>I, b<sub>100</sub> 86°; 72% Br(CF<sub>2</sub>)<sub>4</sub>I, b<sub>130</sub>; and a mixture of 80% n-C<sub>10</sub>F<sub>21</sub>I and 15% n-C<sub>12</sub>F<sub>25</sub>I. A drawing of the apparatus is included.

=> d his

(FILE 'HOME' ENTERED AT 10:32:28 ON 23 JUN 2006)

FILE 'CPLUS' ENTERED AT 10:32:46 ON 23 JUN 2006

L1 1 S POLYFLUOROALKYLETHYL IODIDE  
L2 89 S POLYFLUOROALKYL IODIDE  
L3 5 S L2 AND ETHYLENE  
L4 8 S TETRAFLUOROETHYLENE AND L2  
L5 6 S L4 NOT L3

=> s polyfluoroalkyl acrylate  
1572 POLYFLUOROALKYL  
1 POLYFLUOROALKYLS  
1572 POLYFLUOROALKYL  
(POLYFLUOROALKYL OR POLYFLUOROALKYLS)  
180045 ACRYLATE  
35260 ACRYLATES  
189689 ACRYLATE  
(ACRYLATE OR ACRYLATES)  
L6 72 POLYFLUOROALKYL ACRYLATE  
(POLYFLUOROALKYL (W) ACRYLATE)

=> s 16 and tetrafluoroethylene  
25843 TETRAFLUOROETHYLENE  
34 TETRAFLUOROETHYLENES  
25850 TETRAFLUOROETHYLENE  
(TETRAFLUOROETHYLENE OR TETRAFLUOROETHYLENES)  
L7 1 L6 AND TETRAFLUOROETHYLENE

=> d 17 ibib ab

L7 ANSWER 1 OF 1 CPLUS COPYRIGHT 2006 ACS on STN  
ACCESSION NUMBER: 1993:214334 CPLUS  
DOCUMENT NUMBER: 118:214334  
TITLE: UV absorbers based on fluorine-containing acrylic polymers  
INVENTOR(S): Kodama, Shunichi; Kawasaki, Toru; Kobayashi, Shigeki;  
Funaki, Atsushi  
PATENT ASSIGNEE(S): Asahi Glass Co., Ltd., Japan  
SOURCE: Jpn. Kokai Tokkyo Koho, 5 pp.  
CODEN: JKXXAF  
DOCUMENT TYPE: Patent  
LANGUAGE: Japanese  
FAMILY ACC. NUM. COUNT: 1  
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
JP 04288387	A2	19921013	JP 1991-75681	19910315
JP 2980999	B2	19991122		

PRIORITY APPLN. INFO.: JP 1991-75681 19910315  
AB Title polymers are obtained from radical-polymerizable double bond-containing compds. [for example, (meth)acryloyl group-containing benzophenones, Ph salicylates, or benzotriazoles] and C4-21-polyfluoroalkyl or polyfluoroether-containing (meth)acrylates at mol ratio 90/10 - 10/90 and show weight average mol. weight (Mw) 1000-100,000. Thus, autoclaving 71 parts 2-(3'-hydroxy-4'-benzoyl)phenyloxyethyl methacrylate (I) and 126 parts perfluoroalkylethyl acrylate (II) in Me<sub>2</sub>CO in the presence of peroxybutyl isobutyrate under N at 65° gave a copolymer (III) with Mw .apprx.5000. A 25 μm-thick film extruded from a mixture of 100 parts ethylene-chlorotrifluoroethylene-tetrafluoroethylene copolymer and 5 parts of the III showed UV permeation 0% at 340 nm and 0.2% at 360 nm initially and 0 and 0.3%, resp., after 1000-h exposure to a Sunshine arc weatherometer.

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<input type="checkbox"/>	L12	L11 and ethylene	47
<input type="checkbox"/>	L11	method with (produc\$3 or prepar\$3 or mak\$3)same (polyfluoroalkyl iodide or perfluoroalkyl iodide)	110
<input type="checkbox"/>	L10	L9 not l5 not l6	34
<input type="checkbox"/>	L9	L8 not l1	34
<input type="checkbox"/>	L8	L7 and ethylene	35
<input type="checkbox"/>	L7	process with (produc\$3 or prepar\$3 or mak\$3)same (polyfluoroalkyl iodide or perfluoroalkyl iodide)	109
<input type="checkbox"/>	L6	l5 not l1	12
<input type="checkbox"/>	L5	L4 and tetrafluoroethylene	12
<input type="checkbox"/>	L4	L2 and ethylene	66
<input type="checkbox"/>	L3	L2 and copper with catalyst	1
<input type="checkbox"/>	L2	polyfluoroalkyl acrylate	99
<input type="checkbox"/>	L1	polyfluoroalkylethyl acrylate	2

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<input type="checkbox"/>	L11	method with (produc\$3 or prepar\$3 or mak\$3)same (polyfluoroalkyl iodide or perfluoroalkyl iodide)	110
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<input type="checkbox"/>	L8	L7 and ethylene	35
<input type="checkbox"/>	L7	process with (produc\$3 or prepar\$3 or mak\$3)same (polyfluoroalkyl iodide or perfluoroalkyl iodide)	109
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<input type="checkbox"/>	L3	L2 and copper with catalyst	1
<input type="checkbox"/>	L2	polyfluoroalkyl acrylate	99
<input type="checkbox"/>	L1	polyfluoroalkylethyl acrylate	2

END OF SEARCH HISTORY

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NEWS 5 FEB 22 The IPC thesaurus added to additional patent databases on STN  
NEWS 6 FEB 22 Updates in EPFULL; IPC 8 enhancements added  
NEWS 7 FEB 27 New STN AnaVist pricing effective March 1, 2006  
NEWS 8 MAR 03 Updates in PATDPA; addition of IPC 8 data without attributes  
NEWS 9 MAR 22 EMBASE is now updated on a daily basis  
NEWS 10 APR 03 New IPC 8 fields and IPC thesaurus added to PATDPAFULL  
NEWS 11 APR 03 Bibliographic data updates resume; new IPC 8 fields and IPC thesaurus added in PCTFULL  
NEWS 12 APR 04 STN AnaVist \$500 visualization usage credit offered  
NEWS 13 APR 12 LINSPEC, learning database for INSPEC, reloaded and enhanced  
NEWS 14 APR 12 Improved structure highlighting in FQHIT and QHIT display in MARPAT  
NEWS 15 APR 12 Derwent World Patents Index to be reloaded and enhanced during second quarter; strategies may be affected  
NEWS 16 MAY 10 CA/CAplus enhanced with 1900-1906 U.S. patent records  
NEWS 17 MAY 11 KOREAPAT updates resume  
NEWS 18 MAY 19 Derwent World Patents Index to be reloaded and enhanced  
NEWS 19 MAY 30 IPC 8 Rolled-up Core codes added to CA/CAplus and USPATFULL/USPAT2  
NEWS 20 MAY 30 The F-Term thesaurus is now available in CA/CAplus  
NEWS 21 JUN 02 The first reclassification of IPC codes now complete in INPADOC

NEWS EXPRESS FEBRUARY 15 CURRENT VERSION FOR WINDOWS IS V8.01a,  
CURRENT MACINTOSH VERSION IS V6.0c(ENG) AND V6.0Jc(JP),  
AND CURRENT DISCOVER FILE IS DATED 19 DECEMBER 2005.  
V8.0 AND V8.01 USERS CAN OBTAIN THE UPGRADE TO V8.01a AT  
<http://download.cas.org/express/v8.0-Discover/>

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L1 STRUCTURE UPLOADED

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SAMPLE SEARCH INITIATED 17:55:04 FILE 'CASREACT'

SCREENING COMPLETE - 246 REACTIONS TO VERIFY FROM 25 DOCUMENTS

100.0% DONE 246 VERIFIED 0 HIT RXNS 0 DOCS  
SEARCH TIME: 00.00.01

FULL FILE PROJECTIONS: ONLINE \*\*COMPLETE\*\*  
BATCH \*\*COMPLETE\*\*

PROJECTED VERIFICATIONS: 3980 TO 5860

PROJECTED ANSWERS: 0 TO 0

L2            0 SEA SSS SAM L1 (        0 REACTIONS)

=> s 11 full

FULL SEARCH INITIATED 17:55:17 FILE 'CASREACT'  
SCREENING COMPLETE - 4448 REACTIONS TO VERIFY FROM        437 DOCUMENTS

100.0% DONE    4448 VERIFIED        0 HIT RXNS            0 DOCS  
SEARCH TIME: 00.00.04

L3            0 SEA SSS FUL L1 (        0 REACTIONS)

=> s 11 sss full

FULL SEARCH INITIATED 17:55:36 FILE 'CASREACT'  
SCREENING COMPLETE - 4448 REACTIONS TO VERIFY FROM        437 DOCUMENTS

100.0% DONE    4448 VERIFIED        0 HIT RXNS            0 DOCS  
SEARCH TIME: 00.00.04

L4            0 SEA SSS FUL L1 (        0 REACTIONS)

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FILE LAST UPDATED: 22 Jun 2006 (20060622/ED)

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=> s ethylene and perfluoroalkyl iodide  
524665 ETHYLENE  
3356 ETHYLENES  
526141 ETHYLENE  
    (ETHYLENE OR ETHYLENES)  
10971 PERFLUOROALKYL  
35 PERFLUOROALKYLS  
10991 PERFLUOROALKYL  
    (PERFLUOROALKYL OR PERFLUOROALKYLS)  
173365 IODIDE  
24137 IODIDES  
183154 IODIDE  
    (IODIDE OR IODIDES)

691 PERFLUOROALKYL IODIDE  
(PERFLUOROALKYL(W) IODIDE)  
L5 35 ETHYLENE AND PERFLUOROALKYL IODIDE

=> s 15 and copper  
890360 COPPER  
436 COPPERS  
890423 COPPER  
(COPPER OR COPPERS)

L6 4 L5 AND COPPER

=> d 16 ibib ab 1-4

L6 ANSWER 1 OF 4 CAPLUS COPYRIGHT 2006 ACS on STN

ACCESSION NUMBER: 2004:125104 CAPLUS

DOCUMENT NUMBER: 140:320999

TITLE: Environmentally Benign Processes for Making Useful Fluorocarbons: Nickel- or Copper(I) Iodide-Catalyzed Reaction of Highly Fluorinated Epoxides with Halogens in the Absence of Solvent and Thermal Addition of CF<sub>2</sub>I<sub>2</sub> to Olefins

AUTHOR(S): Yang, Zhen-Yu

CORPORATE SOURCE: Experimental Station, Central Research Development, E. I. Du Pont de Nemours and Co., Wilmington, DE, 19880-0328, USA

SOURCE: Journal of Organic Chemistry (2004), 69(7), 2394-2403  
CODEN: JOCEAH; ISSN: 0022-3263

PUBLISHER: American Chemical Society

DOCUMENT TYPE: Journal

LANGUAGE: English

OTHER SOURCE(S): CASREACT 140:320999

AB Highly fluorinated epoxides react with halogens in the presence of nickel powder or CuI at elevated temps. to provide a useful and general synthesis of dihalodifluoromethanes (CF<sub>2</sub>X<sub>2</sub>) and fluoroacyl fluorides in the absence of solvent. At 185 °C, hexafluoropropylene oxide and halogens produce CF<sub>2</sub>X<sub>2</sub> (X = I, Br) in 68-90% isolated yields, along with small amts. of X(CF<sub>2</sub>)<sub>n</sub>X, (n = 2, 3). With interhalogens I-X (X = Cl, Br), a mixture of CF<sub>2</sub>I<sub>2</sub>, CF<sub>2</sub>XI, and CF<sub>2</sub>X<sub>2</sub> was obtained. The fluorinated epoxides substituted with perfluorophenyl, fluorosulfonyl, and chlorofluoroalkyl groups also react cleanly with iodine to give CF<sub>2</sub>I<sub>2</sub> and the corresponding fluorinated acyl fluorides in good yields. The reaction probably involves an oxidative addition of fluorinated epoxides into metal surfaces to form an oxametallacycle, followed by rapid decomposition to difluorocarbene-metal surfaces, which alters the reactivity of the difluorocarbene carbon from electrophilic to nucleophilic. The increase of nucleophilicity of difluorocarbene facilitates the reaction with electrophilic halogens. CF<sub>2</sub>I<sub>2</sub> reacted with olefins thermally to give 1,3-diiodofluoropropane derivs. Both fluorinated and non-fluorinated alkenes gave good yields of the adducts. Reaction with ethylene, propylene, perfluoroalkylethylene, vinylidene fluoride, and trifluoroethylene provided the corresponding adducts in 58-86% yields. With tetrafluoroethylene, a 1:1 adduct was predominantly formed along with small amts. of higher homologs. In contrast to perfluoroalkyl iodides, CF<sub>2</sub>I<sub>2</sub> also readily adds to perfluorovinyl ethers to give 1,3-diiodoperfluoro ethers. The formation of fluorocarbene was discussed.

REFERENCE COUNT: 117 THERE ARE 117 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L6 ANSWER 2 OF 4 CAPLUS COPYRIGHT 2006 ACS on STN

ACCESSION NUMBER: 1984:591104 CAPLUS

DOCUMENT NUMBER: 101:191104

TITLE: Reactivity of perfluoroiodoalkanes with alkyl carbonates and pyrocarbonates in the presence of a zinc-copper couple  
 AUTHOR(S): Benefice, S.; Blancou, H.; Commeyras, A.  
 CORPORATE SOURCE: Lab. Chim. Org., Univ. Sci. Tech. Languedoc, Montpellier, 34060, Fr.  
 SOURCE: Tetrahedron (1984), 40(9), 1541-4  
 DOCUMENT TYPE: Journal  
 LANGUAGE: French  
 OTHER SOURCE(S): CASREACT 101:191104  
 AB Perfluoroalkyl iodides RI [R = F<sub>3</sub>C(CF<sub>2</sub>)<sub>n</sub>; n = 3, 5, 7] reacted with Zn-Cr in ethylene carbonate at 80-90° to give 60-80% RCO<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>OH, and at >150° to give 60-85% RCO<sub>2</sub>H. RI reacted with Cu-Zn in (EtO)<sub>2</sub>CO or (EtO<sub>2</sub>C)CO to give 50-70, 50-60% RCO<sub>2</sub>Et, resp. In all cases the coupling product RR was also formed.

L6 ANSWER 3 OF 4 CAPLUS COPYRIGHT 2006 ACS on STN  
 ACCESSION NUMBER: 1974:81946 CAPLUS  
 DOCUMENT NUMBER: 80:81946  
 TITLE: Reaction of (perfluoroalkyl)copper compounds with 1-bromo-1-(perfluoroalkyl)ethylenes  
 AUTHOR(S): Santini, G.; Le Blanc, M.; Reiss, J. G.  
 CORPORATE SOURCE: Dep. Chim., Inst. Math. Sci. Phys., Nice, Fr.  
 SOURCE: Tetrahedron (1973), 29(16), 2411-14  
 DOCUMENT TYPE: Journal  
 LANGUAGE: English  
 AB Heating RCB<sub>r</sub>:CH<sub>2</sub> [I, R = (CF<sub>2</sub>)<sub>5</sub>CF<sub>3</sub>, (CF<sub>2</sub>)<sub>7</sub>CF<sub>3</sub>], RII [R = (CF<sub>2</sub>)<sub>3</sub>CF<sub>3</sub>, (CF<sub>2</sub>)<sub>5</sub>CF<sub>3</sub>, (CF<sub>2</sub>)<sub>7</sub>CF<sub>3</sub>], precipitated Cu, and DMF for 20 hr at 120-40° in a sealed tube gave 60-70% trans-RCH:CHR<sub>1</sub>. I were prepared by bromination-dehydrebromination of RCH:CH<sub>2</sub>. <sup>1</sup>H and <sup>19</sup>F NMR were determined

L6 ANSWER 4 OF 4 CAPLUS COPYRIGHT 2006 ACS on STN  
 ACCESSION NUMBER: 1961:33124 CAPLUS  
 DOCUMENT NUMBER: 55:33124  
 ORIGINAL REFERENCE NO.: 55:6496h-i,6497a-c  
 TITLE: Perfluoroalkylated aromatic compounds  
 PATENT ASSIGNEE(S): Minnesota Mining and Manufacturing Co.  
 DOCUMENT TYPE: Patent  
 LANGUAGE: Unavailable  
 FAMILY ACC. NUM. COUNT: 1  
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
GB 840725	---	19600706	GB 1956-24567	19560810
DE 1049862			DE	

AB Aromatic compds. were perfluoroalkylated with the appropriate perfluoroalkyl iodide. Some of the products were converted to dyes. The following reactions were run in a 180 ml. stainless steel rocking autoclave capped by a 3000 lb./sq. in. Ni rupture disk. n-C<sub>3</sub>F<sub>7</sub>I (90 g.) and 23.4 g. C<sub>6</sub>H<sub>6</sub> was heated 15 hrs. at 250°, the crude product filtered off, treated with Hg and distilled to yield 22.3 g. C<sub>3</sub>F<sub>7</sub>Ph, b<sub>760</sub> 132°, n<sub>25D</sub> 1.3790, and 1.5 g. isomeric (C<sub>3</sub>F<sub>7</sub>)<sub>2</sub>C<sub>6</sub>H<sub>4</sub>, n<sub>25D</sub> 1.3492. Likewise prepared were C<sub>7</sub>F<sub>15</sub>Ph (32% yield), b<sub>760</sub> 200°, n<sub>25D</sub> 1.3596, and (C<sub>7</sub>F<sub>15</sub>)<sub>2</sub>C<sub>6</sub>H<sub>4</sub> (4% yield), m. 88°. All subsequent reactions were run in heavy-walled 30 ml. glass ampuls. n-C<sub>7</sub>F<sub>15</sub>I (10 g.) and 0.9 g. PhMe was heated 14 hrs. at 290°, the mixture extracted with perfluorooxacycloheptane and distilled to give a 31% yield of C<sub>7</sub>F<sub>15</sub>C<sub>6</sub>H<sub>4</sub>Me, b<sub>760</sub> 217°, n<sub>25D</sub> 1.3678. Similarly prepared were: C<sub>7</sub>F<sub>15</sub>C<sub>10</sub>H<sub>7</sub> in 31% yield (from naphthalene), n<sub>25D</sub> 1.4160; C<sub>7</sub>F<sub>15</sub>C<sub>6</sub>H<sub>4</sub>CN in 26% yield (from

PhCN), b<sub>760</sub> 254°, m. 45-52°; C<sub>7</sub>F<sub>15</sub>C<sub>6</sub>H<sub>4</sub>Br in 30% yield (from PhBr), b<sub>760</sub> 237°, n<sub>25D</sub> 1.3870; C<sub>7</sub>F<sub>15</sub>C<sub>6</sub>H<sub>4</sub>I in 38% yield (from PhI), b<sub>760</sub> 253°, n<sub>25D</sub> 1.4152; C<sub>7</sub>F<sub>15</sub>C<sub>6</sub>H<sub>3</sub>(CO)2O (I) in 36% yield (from C<sub>6</sub>H<sub>4</sub>(CO)2O), m. 121-4°. I and glycerol in 3:2 mole ratio heated 4 hrs. at 220° gave fluorinated alkyd resins that had varnishlike properties. A perfluoroalkylated dye was prepared as follows: a 30 ml. glass ampul was charged with 1.4 g. Cu phthalocyanine and 10 g. n-C<sub>7</sub>F<sub>15</sub>I. The sealed ampul was heated 14 hrs. at 330°. Extraction with perfluorooxacycloheptane gave 4.4 g. C<sub>6</sub>O<sub>6</sub>F<sub>6</sub>O<sub>12</sub>N<sub>8</sub>Cu (I) (purity approx. 85%). Impurities were removed by vacuum sublimation to yield I, m. 300°, visible absorption at 6550-90 Å. and 6200 Å. I was soluble in fluorinated solvents only. Similarly perfluoroalkylated were the following: thioindigo (Color Index 1207), pyranthrone (Color Index 1096), violanthrone (Color Index 1099), and dichloroisoviolanthrone (Color Index 1104). The principal visible absorption peaks of the products were given. These dyes have been used to dye Teflon.

=> d his

(FILE 'HOME' ENTERED AT 17:54:16 ON 23 JUN 2006)

FILE 'CASREACT' ENTERED AT 17:54:27 ON 23 JUN 2006

L1                   STRUCTURE UPLOADED  
L2                   0 S L1  
L3                   0 S L1 FULL  
L4                   0 S L1 SSS FULL

FILE 'CAPLUS' ENTERED AT 17:56:00 ON 23 JUN 2006

L5                   35 S ETHYLENE AND PERFLUOROALKYL IODIDE  
L6                   4 S L5 AND COPPER

=> s 15 not 16  
L7                   31 L5 NOT L6

=> d 17 ibib ab 1-31

L7   ANSWER 1 OF 31   CAPLUS   COPYRIGHT 2006 ACS on STN  
ACCESSION NUMBER:           2005:735265   CAPLUS  
DOCUMENT NUMBER:           143:220367  
TITLE:                   Method of fabricating dual damascene interconnection  
                         and etchant for stripping sacrificial layer  
INVENTOR(S):           Han, Sang-cheol; Lee, Kyoung-woo; Kim, Mi-young  
PATENT ASSIGNEE(S):       Samsung Electronics Co., Ltd., S. Korea  
SOURCE:                  U.S. Pat. Appl. Publ., 13 pp.  
                          CODEN: USXXCO  
DOCUMENT TYPE:           Patent  
LANGUAGE:                English  
FAMILY ACC. NUM. COUNT:  1  
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
US 2005176243	A1	20050811	US 2005-33208	20050111
PRIORITY APPLN. INFO.:			KR 2004-8065	A 20040206
OTHER SOURCE(S):	MARPAT	143:220367		
AB	A method of forming a dual damascene semiconductor interconnection and an etchant composition specially adapted for stripping a sacrificial layer in a dual damascene fabrication process without profile damage to a dual damascene pattern are provided. The method includes sequentially forming a 1st etch stop layer, a 1st intermetal dielec., a 2nd intermetal dielec., and a capping layer on a surface of a semiconductor substrate on which a			

lower metal wiring is formed; etching the 1st intermetal dielec., the 2nd intermetal dielec., and the capping layer to form a via; forming a sacrificial layer within the via; etching the sacrificial layer, the 2nd intermetal dielec., and the capping layer to form a trench; removing the sacrificial layer remaining around the via using an etchant composition including NH<sub>4</sub>F, HF, H<sub>2</sub>O and a surfactant; and forming an upper metal wiring within the thus formed dual damascene pattern including the via and the trench. The preferred etchant composition for stripping a sacrificial layer in the foregoing dual damascene process consists essentially of NH<sub>4</sub>F, HF, H<sub>2</sub>O and a surfactant.

L7 ANSWER 2 OF 31 CAPLUS COPYRIGHT 2006 ACS on STN

ACCESSION NUMBER: 2005:727519 CAPLUS  
DOCUMENT NUMBER: 144:312852  
TITLE: Amphiphilic block copolymers having water soluble and perfluoroalkyl-group containing blocks  
AUTHOR(S): Kressler, Joerg; Kaiser, Sergej  
CORPORATE SOURCE: Department of Engineering Science Martin-Luther-Universitaet Halle-Wittenberg, Halle, D-06099, Germany  
SOURCE: Polymer Preprints (American Chemical Society, Division of Polymer Chemistry) (2005), 46(2), 580-581  
CODEN: ACPPAY; ISSN: 0032-3934  
PUBLISHER: American Chemical Society, Division of Polymer Chemistry  
DOCUMENT TYPE: Journal; (computer optical disk)  
LANGUAGE: English

AB A series of amphiphilic block copolymer having polyethylene oxide as the water soluble block were synthesized, and then esterified with **perfluoroalkyl iodide**. A second triblock copolymer was synthesized from **ethylene** oxide and hexafluoropropylene oxide. The aggregation behavior of the polymers and the interactions of the above block polymer with lipid monomers were also studied by surface pressure measurement and IRRAS.

REFERENCE COUNT: 7 THERE ARE 7 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L7 ANSWER 3 OF 31 CAPLUS COPYRIGHT 2006 ACS on STN

ACCESSION NUMBER: 2004:769016 CAPLUS  
DOCUMENT NUMBER: 141:395229  
TITLE: An easy three step synthesis of perfluoroalkylated amphetamines  
AUTHOR(S): Tewari, Amit; Hein, Martin; Zapf, Alexander; Beller, Matthias  
CORPORATE SOURCE: Universitaet Rostock, Rostock, 18059, Germany  
SOURCE: Tetrahedron Letters (2004), 45(41), 7703-7707  
CODEN: TELEAY; ISSN: 0040-4039  
PUBLISHER: Elsevier B.V.  
DOCUMENT TYPE: Journal  
LANGUAGE: English  
OTHER SOURCE(S): CASREACT 141:395229

AB A general synthesis of perfluoroalkylated amphetamines is presented. Initially, 1-aryl-1-iodo-2-(perfluoroalkyl)**ethylenes** are prepared by radical addition of **perfluoroalkyl iodides** to arylacetylenes. Key step of the reaction sequence is the following dehydroiodination in the presence of n-BuLi to give 1-perfluoroalkyl-2-arylacetylenes in situ, which are reacted with secondary amines to produce perfluoroalkylated enamines in a new one pot procedure. Final hydrogenation yields the desired products in good yields. By using N,N-dibenzylamine or N-benzylamines the corresponding primary and secondary perfluoroalkylated amines are easily available.

REFERENCE COUNT: 44 THERE ARE 44 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L7 ANSWER 4 OF 31 CAPLUS COPYRIGHT 2006 ACS on STN  
ACCESSION NUMBER: 2004:385293 CAPLUS  
DOCUMENT NUMBER: 141:349790  
TITLE: Pentafluoro- $\lambda$ 6-sulfanyl (SF5)  
**perfluoroalkyl iodides**-synthesis and  
reaction with ethylene and  
tetrafluoroethylene. Crystal structure of  
SF5(CF2)4CH2CH2I  
AUTHOR(S): Nixon, Paul G.; Mohtasham, Javid; Winter, Rolf; Gard,  
Gary L.; Twamley, Brendan; Shreeve, Jean'ne. M.  
CORPORATE SOURCE: Department of Chemistry, Portland State University,  
Portland, OR, 97207-0751, USA  
SOURCE: Journal of Fluorine Chemistry (2004), 125(4), 553-560  
CODEN: JFLCAR; ISSN: 0022-1139  
PUBLISHER: Elsevier Science B.V.  
DOCUMENT TYPE: Journal  
LANGUAGE: English  
OTHER SOURCE(S): CASREACT 141:349790  
AB A series of reactions of SF5CF2CF2I and SF5(CF2)4I with F2C:CF2 was  
carried out in an effort to find the most effective methods for  
chain-extension. SF5(CF2)8I and SF5(CF2)10I were prepared and isolated.  
The reaction conditions for the addition of H2C:CH2 were also investigated.  
The crystal structure of SF5(CF2)4CH2CH2I was determined  
REFERENCE COUNT: 17 THERE ARE 17 CITED REFERENCES AVAILABLE FOR THIS  
RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L7 ANSWER 5 OF 31 CAPLUS COPYRIGHT 2006 ACS on STN  
ACCESSION NUMBER: 2001:292782 CAPLUS  
DOCUMENT NUMBER: 135:121980  
TITLE: A simple procedure for nucleophilic  
perfluoroalkylation of organic and inorganic  
substrates  
AUTHOR(S): Petrov, V. A.  
CORPORATE SOURCE: Experimental Station, Central Research and  
Development, E. I. Du Pont de Nemours and Co.,  
Wilmington, DE, 19880-0328, USA  
SOURCE: Tetrahedron Letters (2001), 42(19), 3267-3269  
CODEN: TELEAY; ISSN: 0040-4039  
PUBLISHER: Elsevier Science Ltd.  
DOCUMENT TYPE: Journal  
LANGUAGE: English  
OTHER SOURCE(S): CASREACT 135:121980  
AB The mixture **perfluoroalkyl iodide** and  
tetrakis(dimethylamino)ethylene is used for the nucleophilic  
perfluoroalkylation. The reaction of chlorotrimethylsilane with  
**perfluoroalkyl iodide** and tetrakis(dimethylamino)  
ethylene in diglyme gives perfluoroalkyltrimethylsilane in 55-81%  
yield. The interaction of this system with organic electrophiles such as  
benzoyl and benzenesulfonyl chlorides, aliphatic and aromatic aldehydes and  
activated ketones leads to the formation of the corresponding condensation  
products in 24-62% yield.  
REFERENCE COUNT: 22 THERE ARE 22 CITED REFERENCES AVAILABLE FOR THIS  
RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L7 ANSWER 6 OF 31 CAPLUS COPYRIGHT 2006 ACS on STN  
ACCESSION NUMBER: 2001:180342 CAPLUS  
DOCUMENT NUMBER: 134:366988  
TITLE: Reactions of Halofluorocarbons with Group 6 Complexes  
M(C5H5)2L (M = Mo, W; L = C2H4, CO). Fluoroalkylation  
at Molybdenum and Tungsten, and at Cyclopentadienyl or  
**Ethylene** Ligands

AUTHOR(S): Hughes, Russell P.; Maddock, Susan M.; Guzei, Ilia A.;  
Liable-Sands, Louise M.; Rheingold, Arnold L.  
CORPORATE SOURCE: Departments of Chemistry Burke Chemistry Laboratory,  
Dartmouth College, Hanover, NH, 03755-3564, USA  
SOURCE: Journal of the American Chemical Society (2001),  
123(14), 3279-3288  
CODEN: JACSAT; ISSN: 0002-7863  
PUBLISHER: American Chemical Society  
DOCUMENT TYPE: Journal  
LANGUAGE: English  
OTHER SOURCE(S): CASREACT 134:366988

AB The Mo(II) and W(II) complexes [MCp<sub>2</sub>L] (Cp =  $\eta^5$ -cyclopentadienyl; L = C<sub>2</sub>H<sub>4</sub>, CO) react with **perfluoroalkyl iodides** to give a variety of products. The Mo(II) complex [MoCp<sub>2</sub>(C<sub>2</sub>H<sub>4</sub>)] reacts with perfluoro-BuI or perfluorobenzyl iodide with loss of **ethylene** to give the 1st examples of fluoroalkyl complexes of Mo(IV), MoCp<sub>2</sub>(CF<sub>2</sub>CF<sub>2</sub>CF<sub>3</sub>)I (8) and MoCp<sub>2</sub>(CF<sub>2</sub>C<sub>6</sub>F<sub>5</sub>)I (9), one of which (8) was crystallog. characterized. In contrast, the CO analog [MoCp<sub>2</sub>(CO)] reacts with perfluorobenzyl iodide without loss of CO to give the crystallog. characterized salt, [MoCp<sub>2</sub>(CF<sub>2</sub>C<sub>6</sub>F<sub>5</sub>)(CO)]<sup>+</sup>I<sup>-</sup> (10), and the W(II) **ethylene** precursor [WCp<sub>2</sub>(C<sub>2</sub>H<sub>4</sub>)] reacts with perfluorobenzyl iodide without loss of **ethylene** to afford [WCp<sub>2</sub>(CF<sub>2</sub>C<sub>6</sub>F<sub>5</sub>)(C<sub>2</sub>H<sub>4</sub>)]<sup>+</sup>I<sup>-</sup> (11). These observations demonstrate that the metal-C bond is formed 1st. In further contrast the W precursor [WCp<sub>2</sub>(C<sub>2</sub>H<sub>4</sub>)] reacts with perfluoro-BuI, perfluoro-iso-Pr iodide, and pentafluorophenyl iodide to give fluoroalkyl- and fluorophenyl-substituted cyclopentadienyl complexes WCp( $\eta^5$ -C<sub>5</sub>H<sub>4</sub>RF)(H)I (12, RF = CF<sub>2</sub>CF<sub>2</sub>CF<sub>2</sub>CF<sub>3</sub>; 15, RF = CF(CF<sub>3</sub>)<sub>2</sub>; 16, RF = C<sub>6</sub>F<sub>5</sub>); the Mo analog MoCp( $\eta^5$ -C<sub>5</sub>H<sub>4</sub>RF)(H)I (14, RF = CF(CF<sub>3</sub>)<sub>2</sub>) was obtained in similar fashion. The W(IV) hydrido compds. react with iodoform to afford the corresponding diiodides WCp( $\eta^5$ -C<sub>5</sub>H<sub>4</sub>RF)I<sub>2</sub> (13, RF = CF<sub>2</sub>CF<sub>2</sub>CF<sub>2</sub>CF<sub>3</sub>; 18, RF = CF(CF<sub>3</sub>)<sub>2</sub>; 19, RF = C<sub>6</sub>F<sub>5</sub>), two of which (13 and 19) were crystallog. characterized. The carbonyl precursors [MCp<sub>2</sub>(CO)] each react with perfluoro-iso-Pr iodide without loss of CO, to afford the exo-fluoroalkylated cyclopentadiene M(II) complexes MCp( $\eta^4$ -C<sub>5</sub>H<sub>5</sub>RF)(CO)I (21, M = Mo; 22, M = W); the exo-stereochem. for the fluoroalkyl group is confirmed by an x-ray structural study of 22. The **ethylene** analogs [MCp<sub>2</sub>(C<sub>2</sub>H<sub>4</sub>)] react with perfluoro-tert-BuI to yield the products MCp<sub>2</sub>[CH<sub>2</sub>CH<sub>2</sub>C(CF<sub>3</sub>)<sub>3</sub>]I (25, M = Mo; 26, M = W) resulting from fluoroalkylation at the **ethylene** ligand. Attempts to provide pos. evidence for fluoroalkyl radicals as intermediates in reactions of primary and benzylic substrates were unsuccessful, but trapping expts. with CH<sub>3</sub>OD (to give RFD, not RFH) indicate that fluoroalkyl anions are the intermediates responsible for ring and **ethylene** fluoroalkylation in the reactions of secondary and tertiary fluoroalkyl substrates.

REFERENCE COUNT: 65 THERE ARE 65 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L7 ANSWER 7 OF 31 CAPLUS COPYRIGHT 2006 ACS on STN  
ACCESSION NUMBER: 2000:200696 CAPLUS  
DOCUMENT NUMBER: 133:17879  
TITLE: Controlled step-wise telomerization of vinylidene fluoride, hexafluoropropene and trifluoroethylene with iodofluorinated transfer agents  
AUTHOR(S): Balague, J.; Ameduri, B.; Boutevin, B.; Caporiccio, G.  
CORPORATE SOURCE: Ecole Nationale Supérieure de Chimie, UMR 5076 (CNRS),  
Laboratory of Macromolecular Chemistry, Montpellier,  
34296, Fr.  
SOURCE: Journal of Fluorine Chemistry (2000), 102(1-2),  
253-268  
CODEN: JFLCAR; ISSN: 0022-1139  
PUBLISHER: Elsevier Science S.A.

DOCUMENT TYPE:

Journal

LANGUAGE:

English

AB Highly fluorinated cotelomers having the structure F(TFE)<sub>w</sub>(VDF)<sub>x</sub>(HFP)<sub>y</sub>(TrFE)<sub>z</sub>I containing one or several tetrafluoroethylene (TFE), vinylidene fluoride (or 1,1-difluoroethylene, VDF), hexafluoropropene (HFP) or trifluoroethylene (TrFE) base units were synthesized by thermal step-wise cotelomerization of these fluoroolefins with **perfluoroalkyl iodides**. <sup>1</sup>H and <sup>19</sup>F NMR allowed one to characterize these cotelomers and to assess the defects of chaining and the mol. wts. While the monoadduct produced from VDF exclusively exhibits RFCH<sub>2</sub>CF<sub>2</sub>I structure, that prepared from TrFE was composed of RFCFHC<sub>2</sub>I and RFCF<sub>2</sub>CFHI isomers, the ratio of which is directed from the electrophilicity of RF<sup>+</sup> radical. The reactivity of the C-I bond in RF-Q-CXY-I depends on the nature of the Q spacer and on the reactivity of the fluorinated monomer (e.g., thermal initiations of VDF, TrFE and HFP were efficient from 180°, 195° and 210°, resp.). The mechanism of the addition of the radical generated from the iodinated transfer agent to the fluoroalkene is explained by means of its electrophilic attack to the more nucleophilic (i.e., the less electrophilic) side of the olefin. Ethylenation of these fluorocotelomers was successfully achieved from various initiations (thermal, redox or from peroxides) with best results from redox catalysis. Thermal properties of several fluorotelomers (glass transition temps., T<sub>g</sub> and melting temps., T<sub>m</sub>) were assessed. They were linked to the number of consecutive CF<sub>2</sub> groups (for the crystalline zones) and bulky side groups which induced amorphous regions.

REFERENCE COUNT:

80 THERE ARE 80 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L7 ANSWER 8 OF 31 CAPLUS COPYRIGHT 2006 ACS on STN

ACCESSION NUMBER: 2000:200694 CAPLUS

DOCUMENT NUMBER: 133:17205

TITLE: Diododifluoromethane: an excellent telogen for the preparation of 1,3-diodofluoropropane derivatives

AUTHOR(S): Yang, Z.-Y.

CORPORATE SOURCE: Experimental Station, Central Research & Development, E. I. Du Pont de Nemours and Co., Wilmington, DE, USA

SOURCE: Journal of Fluorine Chemistry (2000), 102(1-2), 239-241

PUBLISHER: Elsevier Science S.A.

DOCUMENT TYPE: Journal

LANGUAGE: English

AB CF<sub>2</sub>I<sub>2</sub> reacted with olefins thermally to give 1,3-diodofluoropropane derivs. Both fluorinated and non-fluorinated alkenes gave good yields of adducts. Reaction with **ethylene**, propylene, perfluoroalkylethylene, vinylidene fluoride, and trifluoroethylene provided the corresponding adducts in 58-86% yields. With tetrafluoroethylene, 1-to-1 adduct was predominantly formed along with small amts. of higher homologs. In contrast to **perfluoroalkyl iodides**, CF<sub>2</sub>I<sub>2</sub> also readily added to perfluorovinyl ethers to give 1,3-diiodoperfluoroethers.

REFERENCE COUNT:

29 THERE ARE 29 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L7 ANSWER 9 OF 31 CAPLUS COPYRIGHT 2006 ACS on STN

ACCESSION NUMBER: 1997:403224 CAPLUS

DOCUMENT NUMBER: 127:81545

TITLE: Selective Fluoroalkylation of Cyclopentadienyl and **Ethylene Ligands** in Reactions of **Perfluoroalkyl Iodides** with Low-Valent Complexes of Molybdenum and Tungsten:

AUTHOR(S): Evidence for a Fluorocarbanion Mechanism  
 Hughes, Russell P.; Maddock, Susan M.; Rheingold,  
 Arnold L.; Liable-Sands, Louise M.  
 CORPORATE SOURCE: Department of Chemistry 6128 Burke Laboratory,  
 Dartmouth College, Hanover, NH, 03755, USA  
 SOURCE: Journal of the American Chemical Society (1997),  
 119(25), 5988-5989  
 CODEN: JACSAT; ISSN: 0002-7863  
 PUBLISHER: American Chemical Society  
 DOCUMENT TYPE: Journal  
 LANGUAGE: English  
 OTHER SOURCE(S): CASREACT 127:81545  
 AB Reactions of **perfluoroalkyl iodide**  $\text{ICF}(\text{CF}_3)_2$  with  
 $[\text{MCp}_2(\text{C}_2\text{H}_4)]$  (2, M = Mo; 3, M = W) afford the hydrido complexes  
 $[\{\text{C}_5\text{H}_4\text{CF}(\text{CF}_3)_2\}(\text{Cp})]\text{MHI}$  (4, M = Mo; 5, M = W) quant. The structure of 5  
 has been confirmed by x-ray crystallog.; monoclinic,  $P\bar{1}/c$ ,  $a = 6.268(2)$ ,  
 $b = 33.748(6)$ ,  $c = 7.616(2)$  Å,  $\beta = 112.01(2)^\circ$ ,  $V = 1493.6(6)$  Å<sup>3</sup>,  $Z = 4$ . In contrast, the tertiary **perfluoroalkyl iodide**  $\text{IC}(\text{CF}_3)_3$  reacts with 2 or 3 cleanly at the ethylene  
 ligand to give 6 and 7. X-ray crystallog. also confirms the structure of 7; monoclinic,  $P21/n$ ,  $a = 12.8936(1)$ ,  $b = 7.557(1)$ ,  $c = 38.9142(2)$  Å,  
 $\beta = 95.0237(2)^\circ$ ,  $V = 3777.46(6)$  Å<sup>3</sup>,  $Z = 8$  (two chemical  
 identical, crystallog. independent mols.). Running these reactions in the  
 presence of radical traps like perdeuterotoluene or dihydroanthraene  
 provides no evidence for fluoroalkyl radical intermediates, but pos.  
 signatures for fluorocarbanion intermediates are provided by trapping  
 expts. with  $\text{CH}_3\text{OD}$ , and by observation of fluoroolefins  $\text{F}_2\text{C:CF}(\text{CF}_3)$  and  
 $\text{F}_2\text{C:C}(\text{CF}_3)_2$  among the reaction products from  $\text{ICF}(\text{CF}_3)_2$  or  $\text{IC}(\text{CF}_3)_3$  resp.  
 REFERENCE COUNT: 27 THERE ARE 27 CITED REFERENCES AVAILABLE FOR THIS  
 RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L7 ANSWER 10 OF 31 CAPLUS COPYRIGHT 2006 ACS on STN  
 ACCESSION NUMBER: 1995:650376 CAPLUS  
 DOCUMENT NUMBER: 123:256153  
 TITLE: Preparation of **perfluoroalkyl iodides** from perfluorocarboxylic acids  
 INVENTOR(S): Ueda, Kunimasa  
 PATENT ASSIGNEE(S): Idemitsu Petrochemical Co, Japan  
 SOURCE: Jpn. Kokai Tokyo Koho, 12 pp.  
 CODEN: JKXXAF  
 DOCUMENT TYPE: Patent  
 LANGUAGE: Japanese  
 FAMILY ACC. NUM. COUNT: 1  
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
JP 07109234	A2	19950425	JP 1993-277348	19931012
PRIORITY APPLN. INFO.:			JP 1993-277348	19931012

OTHER SOURCE(S): MARPAT 123:256153  
 AB In preparation of  $\text{C}_n\text{F}_m\text{I}$  ( $n \geq 1$ ;  $3 \leq m \leq 2n + 1$ ), useful as  
 perfluoroalkylating agents by treatment of  $\text{C}_n\text{F}_m\text{CO}_2\text{H}$  with I and  $\text{Bz}_2\text{O}_2$  (I)  
 in an organic solvent, a homogeneous solution or suspension of I in the organic  
 solvent, obtained by treatment of I containing  $\text{H}_2\text{O}$  with the organic solvent,  
 which is suitable for the above iodination and practically immiscible with  
 $\text{H}_2\text{O}$ , to extract I followed by separation and removal of the aqueous layer, is  
 used as  
 I and the solvent. The method is prevented from danger of explosive I. I  
 containing 25 weight%  $\text{H}_2\text{O}$  was suspended in  $\text{Cl}(\text{CH}_2)_4\text{Cl}$  (II) and the upper  $\text{H}_2\text{O}$   
 layer was removed. The suspension was treated with  $\text{CF}_3(\text{CF}_2)_7\text{CO}_2\text{H}$ , II, and  
 I under reflux at  $100^\circ$  for 1 h to give 77%  $\text{CF}_3(\text{CF}_2)_7\text{I}$ .

L7 ANSWER 11 OF 31 CAPLUS COPYRIGHT 2006 ACS on STN  
 ACCESSION NUMBER: 1995:321022 CAPLUS  
 DOCUMENT NUMBER: 123:313360  
 TITLE: Free-radical addition of 2-(perfluoroalkyl)ethanethiols to alkenes, alkadienes, cycloalkenes, alkynes and vinyl monomers. [Erratum to document cited in CA119:225554]  
 AUTHOR(S): Brace, Neal O.  
 CORPORATE SOURCE: Wheaton College, Wheaton, IL, 60187, USA  
 SOURCE: Journal of Fluorine Chemistry (1995), 70(1), 145  
 CODEN: JFLCAR; ISSN: 0022-1139  
 PUBLISHER: Elsevier  
 DOCUMENT TYPE: Journal  
 LANGUAGE: English  
 AB The errors were not reflected in the abstract or the index entries.

L7 ANSWER 12 OF 31 CAPLUS COPYRIGHT 2006 ACS on STN  
 ACCESSION NUMBER: 1994:136059 CAPLUS  
 DOCUMENT NUMBER: 120:136059  
 TITLE: Perfluoroalkyl halides and derivatives as precursors for oil and water repellants and surfactants  
 INVENTOR(S): Behr, Frederick E.; Dams, Rudolf J.; DeWitte, Johan E.; Hagen, Donald F.  
 PATENT ASSIGNEE(S): Minnesota Mining and Manufacturing Co., USA  
 SOURCE: Can. Pat. Appl., 67 pp.  
 CODEN: CPXXEB  
 DOCUMENT TYPE: Patent  
 LANGUAGE: English  
 FAMILY ACC. NUM. COUNT: 3  
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
CA 2071596	AA	19930111	CA 1992-2071596	19920618
EP 526976	A1	19930210	EP 1992-305710	19920622
EP 526976	B1	19970115		
R: BE, CH, DE, FR, GB, IT, LI, NL				
JP 05345732	A2	19931227	JP 1992-183345	19920710
JP 3231844	B2	20011126		
JP 2002138078	A2	20020514	JP 2001-204928	19920710
PRIORITY APPLN. INFO.:			US 1991-728184	A 19910710
			JP 1992-183345	A3 19920710

OTHER SOURCE(S): MARPAT 120:136059

AB The title compds. comprise a mixture of straight and branched perfluoroalkyl groups bonded to Cl, Br, or I through a F-free alkylene group. Perfluorodecyltetrahydroiodide (prepared from perfluorosulfonyl fluoride, 40% straight and 60% branched, treated first with I, then with C<sub>2</sub>H<sub>4</sub>) was derivatized to thiol functionality by treatment with thiourea in EtOH to give perfluorodecyltetrahydrothiol (I). I was added to a reaction mixture containing hexamethoxymethylmelamine to give a I-melamine condensate (II, 1:4 mol ratio). A 50/50 polyester/cotton fabric blend was treated with an emulsion of II at 0.3%, dried and cured at 150°, to give a fabric with oil resistance (AATCC 118-1975) 5 and 5 after 1 dry cleaning, vs. 3 and 2, resp., for a precursor perfluorodecyltetrahydroiodide having all straight chain perfluoroalkyl groups.

L7 ANSWER 13 OF 31 CAPLUS COPYRIGHT 2006 ACS on STN  
 ACCESSION NUMBER: 1993:625554 CAPLUS  
 DOCUMENT NUMBER: 119:225554  
 TITLE: Free-radical addition of 2-(perfluoroalkyl)ethanethiols to alkenes, alkadienes, cycloalkenes, alkynes and vinyl monomers

AUTHOR(S): Brace, Neal O.  
 CORPORATE SOURCE: Wheaton Coll., Wheaton, IL, 60187, USA  
 SOURCE: Journal of Fluorine Chemistry (1993), 62(2-3), 217-41  
 CODEN: JFLCAR; ISSN: 0022-1139  
 DOCUMENT TYPE: Journal  
 LANGUAGE: English  
 AB The free-radical addition of 2-(perfluoroalkyl)ethanethiols ( $\text{RFCH}_2\text{CH}_2\text{SH}$ ) to alkenes, cycloalkenes, alkadienes and alkynes has been studied to determine (1) the mode of reaction, i.e., the stereochem., regiochem. and any skeletal changes; (2) the relative reactivity towards unsats. of various structures and classes as affected by the presence of the RF group; and (3) the influence of the reaction conditions on the rate of addition or selectivity for different products. Adducts from 2-(F-hexyl)ethanethiol (1) and alkenes have been obtained in high yield, but containing small amts. of regioisomers. For example, compound 1 with 1-heptene gave 96% 1-[2-(F-hexyl)ethanethio]heptane, as well as 0.61% 2- and 2.22% 3-[2-(F-hexyl)ethanethio]heptane. Dienes gave chiefly linear adducts adducts; small amts. of cyclic isomers were also formed. Compound 1 added readily with free-radical initiation to vinyl monomers such as styrene and vinyl acetate, and to phenylacetylene, propargyl acetate and Et propynoate.

L7 ANSWER 14 OF 31 CAPLUS COPYRIGHT 2006 ACS on STN  
 ACCESSION NUMBER: 1988:454260 CAPLUS  
 DOCUMENT NUMBER: 109:54260  
 TITLE: Synthesis and chemistry of perfluoro-2-iodo-2-methylalkanes  
 AUTHOR(S): Probst, A.; Raab, K.; Ulm, K.; Von Werner, K.  
 CORPORATE SOURCE: Werk Gendorf, Hoechst A.-G., Burgkirchen, D-8269, Fed. Rep. Ger.  
 SOURCE: Journal of Fluorine Chemistry (1987), 37(2), 223-45  
 CODEN: JFLCAR; ISSN: 0022-1139  
 DOCUMENT TYPE: Journal  
 LANGUAGE: English  
 OTHER SOURCE(S): CASREACT 109:54260  
 AB  $\text{CF}_3(\text{CF}_2)_n\text{C}(\text{CF}_3)_2\text{I}$  ( $I$ ,  $n = 1, 2$ ) were obtained from  $\text{CF}_3(\text{CF}_2)_n\text{CF:C}(\text{CF}_3)_2$  ( $n = 0, 1$ ) by formal addns. of IF that required substantial alterations of known procedures. I ( $n = 1, 2$ ) are the most reactive alkyl halides known so far, and they are also very toxic. The reactions studied included (a) Nucleophilic attack of anions at the iodine, leading to perfluoroalkenes, (b) elimination of IF, caused by metals or metal complexes, (c) pyrolysis, to selectively give perfluoroisobutene and n-perfluoroalkyl iodides, (d) photolysis, and (e) thermally induced insertions into the carbon-iodine bond. Screening results on the inhalation toxicity of the iodides and of some other fluoro-compds. are also reported.

L7 ANSWER 15 OF 31 CAPLUS COPYRIGHT 2006 ACS on STN  
 ACCESSION NUMBER: 1987:33621 CAPLUS  
 DOCUMENT NUMBER: 106:33621  
 TITLE:  $\omega$ -Perfluoroalkyl- $\alpha$ -olefin polymers  
 INVENTOR(S): Su, Aaron Chung Liang  
 PATENT ASSIGNEE(S): du Pont de Nemours, E. I., and Co., USA  
 SOURCE: Eur. Pat. Appl., 18 pp.  
 CODEN: EPXXDW  
 DOCUMENT TYPE: Patent  
 LANGUAGE: English  
 FAMILY ACC. NUM. COUNT: 1  
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
EP 193202	A2	19860903	EP 1986-102650	19860228

EP 193202	A3	19870722		
R: DE, FR, GB, IT				
US 4617363	A	19861014	US 1985-707013	19850228
CA 1275544	A1	19901023	CA 1986-502693	19860225
JP 61204208	A2	19860910	JP 1986-42027	19860228
JP 03080804	B4	19911226		
JP 01103609	A2	19890420	JP 1988-208460	19880824

PRIORITY APPLN. INFO.:

AB Polymers of CH<sub>2</sub>:CH(CH<sub>2</sub>)<sub>n</sub>R (R = C<sub>2</sub>-10 perfluoroalkyl; n = 2-8) have high gas-selective permeability and solvent resistance. Thus, 20 mL C<sub>4</sub>F<sub>9</sub>(CH<sub>2</sub>)<sub>2</sub>CH:CH<sub>2</sub> (prepared by addition of C<sub>4</sub>F<sub>9</sub>(CH<sub>2</sub>)<sub>2</sub>I and C<sub>2</sub>H<sub>4</sub>, then dehydroiodination with tert-BuOK), 3 mmol iso-Bu<sub>3</sub>Al, and 0.3 g TiCl<sub>4</sub> on MgCl<sub>2</sub> were heated at 50° for 4 h to form poly(4-perfluorobutyl-1-butene) having 100% modulus 3.4 MPa and tensile strength 10.3 MPa, which was insol. in hydrocarbon, fluorocarbon, and polar solvents, and showed CO<sub>2</sub> permeability (80:20 CO<sub>2</sub>/CH<sub>4</sub>) 7.7 + 10<sup>-8</sup> cm<sup>3</sup>-cm/cm<sup>2</sup>-s-cm Hg and selectivity [(CO<sub>2</sub>/CH<sub>4</sub> in product)/(CO<sub>2</sub>/CH<sub>4</sub> in feed)] 9; vs. 1.2 + 10<sup>-9</sup> cm<sup>3</sup>-cm/cm<sup>2</sup>-s-cm Hg and 10 for Teflon; or 5 + 10<sup>-8</sup> cm<sup>3</sup>-cm/cm<sup>2</sup>-s-cm Hg and 5 for natural rubber.

L7 ANSWER 16 OF 31 CAPLUS COPYRIGHT 2006 ACS on STN

ACCESSION NUMBER: 1987:17597 CAPLUS  
 DOCUMENT NUMBER: 106:17597  
 TITLE: Fluoroiodo compounds  
 PATENT ASSIGNEE(S): du Pont de Nemours, E. I., and Co., USA  
 SOURCE: Jpn. Kokai Tokkyo Koho, 11 pp.  
 CODEN: JKXXAF  
 DOCUMENT TYPE: Patent  
 LANGUAGE: Japanese  
 FAMILY ACC. NUM. COUNT: 1  
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
JP 61134329	A2	19860621	JP 1985-269194	19851129
US 4650913	A	19870317	US 1984-676100	19841129
EP 194348	A2	19860917	EP 1985-115132	19851129
EP 194348	A3	19870819		
EP 194348	B1	19900124		

R: CH, DE, FR, LI

PRIORITY APPLN. INFO.: US 1984-676100 A 19841129

OTHER SOURCE(S): CASREACT 106:17597; MARPAT 106:17597

AB RCR<sub>1</sub>R<sub>2</sub>CR<sub>3</sub>R<sub>4</sub>I (R = C<sub>1</sub>-18 perfluorohydrocarbon residue; R<sub>1-4</sub> = H, C<sub>1</sub>-16 hydrocarbon, mono- or bicyclic aliphatic residue) are prepared by addition of R<sub>1</sub>R<sub>2</sub>C:CRR<sub>4</sub> with RI in the presence of R<sub>5</sub>SO<sub>2</sub>M (R<sub>5</sub> = C<sub>1</sub>-18 hydrocarbon residue, M = alkali metal, NH<sub>4</sub>, etc.) as initiators. Thus a mixture of norbornene 0.1, CF<sub>3</sub>(CF<sub>2</sub>)<sub>7</sub>I 0.02, and PhSO<sub>2</sub>Na 0.03 mol in DMF was stirred under Ar to give 93% 2-iodo-3-(perfluorooctyl)norbornane.

L7 ANSWER 17 OF 31 CAPLUS COPYRIGHT 2006 ACS on STN

ACCESSION NUMBER: 1985:614859 CAPLUS  
 DOCUMENT NUMBER: 103:214859  
 TITLE: Fluoroalkyl-substituted iodoalkanes  
 INVENTOR(S): Von Werner, Konrad  
 PATENT ASSIGNEE(S): Hoechst A.-G. , Fed. Rep. Ger.  
 SOURCE: Ger. Offen., 19 pp.  
 CODEN: GWXXBX  
 DOCUMENT TYPE: Patent  
 LANGUAGE: German  
 FAMILY ACC. NUM. COUNT: 1  
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
DE 3338300	A1	19850502	DE 1983-3338300	19831021
EP 140254	A1	19850508	EP 1984-112300	19841012
EP 140254	B1	19881005		
R: BE, CH, DE, FR, GB, IT, LI, NL				
JP 60106533	A2	19850612	JP 1984-218786	19841019
US 4587366	A	19860506	US 1984-663084	19841019
PRIORITY APPLN. INFO.:			DE 1983-3338300	A 19831021

OTHER SOURCE(S): MARPAT 103:214859

AB (Fluoroalkyl)alkyl iodides were prepared by reaction of a fluorinated alkyl iodide with warm (un)substituted alkenes in the presence of a finely-divided metal catalyst chosen from elements with atomic nos. 24-30, 42-48, or 74-79. I(CF<sub>2</sub>)<sub>4</sub>I (0.68 mol) reacted with G0.68 mol CH<sub>2</sub>:CH<sub>2</sub> in the presence of Ru at 170°/2 MPa in 33 h to give 97.8% yield of I(CH<sub>2</sub>)<sub>2</sub>(CF<sub>2</sub>)<sub>4</sub>(CH<sub>2</sub>)<sub>2</sub>I with 98.5% conversion I(CF<sub>2</sub>)<sub>4</sub>I.

L7 ANSWER 18 OF 31 CAPLUS COPYRIGHT 2006 ACS on STN

ACCESSION NUMBER: 1984:51074 CAPLUS  
 DOCUMENT NUMBER: 100:51074  
 TITLE: Branched 1,2-bis(perfluoroalkyl)ethenes  
 INVENTOR(S): Riess, Jean Georges; Jeanneaux, Francois; Le Blanc, Maurice; Lantz, Andre  
 PATENT ASSIGNEE(S): Produits Chimiques Ugine Kuhlmann, Fr.  
 SOURCE: Eur. Pat. Appl., 12 pp.  
 CODEN: EPXXDW  
 DOCUMENT TYPE: Patent  
 LANGUAGE: French  
 FAMILY ACC. NUM. COUNT: 1  
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
EP 90712	A1	19831005	EP 1983-400569	19830318
EP 90712	B1	19850619		
R: AT, BE, CH, DE, FR, GB, IT, LI, NL, SE				
FR 2523956	A1	19830930	FR 1982-5165	19820326
FR 2523956	B1	19850524		
US 4613708	A	19860923	US 1983-467648	19830218
AT 13872	E	19850715	AT 1983-400569	19830318
AU 8312872	A1	19830929	AU 1983-12872	19830325
AU 554646	B2	19860828		
JP 58174334	A2	19831013	JP 1983-49089	19830325
JP 01055251	B4	19891122		
ES 521022	A1	19840601	ES 1983-521022	19830325
CA 1186344	A1	19850430	CA 1983-424516	19830325
PRIORITY APPLN. INFO.:			FR 1982-5165	A 19820326
			EP 1983-400569	A 19830318

OTHER SOURCE(S): CASREACT 100:51074; MARPAT 100:51074

AB Perfluoroalkyl iodides reacted with mono(perfluoroalkyl)ethylenes, and the addition products obtained were dehydriodinated to yield RCH:CHR<sub>1</sub> (R and R<sub>1</sub> are perfluoroalkyl and one or both of them is/are branched), useful in emulsions. A mixture of CH<sub>2</sub>:CHCF(CF<sub>3</sub>)<sub>2</sub> and (CF<sub>3</sub>)<sub>2</sub>CFI was heated at 200° to give (CF<sub>3</sub>)<sub>2</sub>CFCH<sub>2</sub>CHCF(CF<sub>3</sub>)<sub>2</sub>, which was treated with KOH in EtOH to give (CF<sub>3</sub>)<sub>2</sub>CFCH:CHCF(CF<sub>3</sub>)<sub>2</sub>.

L7 ANSWER 19 OF 31 CAPLUS COPYRIGHT 2006 ACS on STN

ACCESSION NUMBER: 1982:217164 CAPLUS  
 DOCUMENT NUMBER: 96:217164  
 TITLE: Synthesis of fluorinated acetylenes  
 AUTHOR(S): Baum, Kurt; Bedford, Clifford D.; Hunad, Ronald J.

CORPORATE SOURCE: Fluorochem, Inc., Azusa, CA, 91702, USA  
SOURCE: Journal of Organic Chemistry (1982), 47(12), 2251-7  
CODEN: JOCEAH; ISSN: 0022-3263  
DOCUMENT TYPE: Journal  
LANGUAGE: English  
OTHER SOURCE(S): CASREACT 96:217164

AB New routes to fluorinated acetylenes were developed based on addns. of iodofluorocarbons to silylacetylenes. Free radical addition of  $\alpha$ -diodoperfluoroalkanes to (trimethylsilyl)acetylene gave the iodotrimethylsilyl olefins  $\text{Me}_3\text{SiIC:CH(CF}_2\text{nCH:CISiMe}_3$ , which reacted with  $\text{KOCMe}_3$  or 1,8-diazabicyclo[5.4.0]undec-7-ene to give (trimethylsilyl)acetylenes and, with an excess of the base, the free diacetylenes. **Perfluoroalkyl iodides** similarly gave (perfluoroalkyl)acetylenes. The addition of perfluoroheptyl iodide to PhC.tpbond.CH followed by treatment with  $\text{KOCMe}_3$  gave 1-phenylperfluorononyne. The peroxide-catalyzed reaction of **perfluoroalkyl iodides** (RFI) and  $\text{Me}_3\text{SiC.tpbond.CSiMe}_3$  gave 1:1 adducts,  $\text{RF(Me}_2\text{SiCH}_2\text{)IC:CHSiMe}_3$ , resulting from intramol. H abstraction by the initially formed vinyl radical. The thermal reaction of **perfluoroalkyl iodides** and diiodides with bis(trimethylsilyl)acetylene in the presence of free iodine gave the (trimethylsilyl)acetylenes, which were desilylated with KF. A route to diacetylenes was investigated based on addition of perfluoroiodo compds. to **ethylene**, dehydroiodination, brominations, and eliminations.

L7 ANSWER 20 OF 31 CAPLUS COPYRIGHT 2006 ACS on STN  
ACCESSION NUMBER: 1978:512054 CAPLUS  
DOCUMENT NUMBER: 89:112054  
TITLE: Modification of fiber surfaces by monomeric additives.  
Part II. Absorption of fluorocarbon additives by poly(**ethylene terephthalate**)  
AUTHOR(S): Mares, F.; Oxenrider, B. C.  
CORPORATE SOURCE: Chem. Res. Cent., Allied Chem. Corp., Morristown, NJ,  
USA  
SOURCE: Textile Research Journal (1978), 48(4), 218-29  
DOCUMENT TYPE: Journal  
LANGUAGE: English  
AB Evaluation of 3 different methods for the modification of poly(**ethylene terephthalate**) fiber surfaces showed that coating the fibers with reactive group-containing additives from a dioxane solution followed by annealing produces the best results. The additives, e.g., an isomeric mixture of dihydrogen bis(4-perfluoroisopropoxy-3,3,4,4-tetrafluorobutyl pyromellitates and mono(4-perfluoroisopropoxy-3,3,4,4-tetrafluorobutyl) phthalate [62478-07-3], were not simply deposited on the fiber surfaces, as demonstrated by scanning electron microscopy, but penetrated the fiber and interacted with the polymer matrix to form oligomers near the fiber surface. Additive concns. as low as 0.25% produced excellent water and oil repellency that was durable to washing and dry cleaning owing to the insol. of the additives in soap solns. and dry cleaning solvents. The modified fibers could be knitted and dyed without affecting the surface properties.

L7 ANSWER 21 OF 31 CAPLUS COPYRIGHT 2006 ACS on STN  
ACCESSION NUMBER: 1978:74021 CAPLUS  
DOCUMENT NUMBER: 88:74021  
TITLE: Addition to gaseous nonhalogenated olefins and acetylenes of **perfluoroalkyl iodides**  
INVENTOR(S): Knell, Martin  
PATENT ASSIGNEE(S): Ciba-Geigy Corp., USA  
SOURCE: U.S., 7 pp.

CODEN: USXXAM

DOCUMENT TYPE:

Patent

LANGUAGE:

English

FAMILY ACC. NUM. COUNT:

3

## PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
US 4058573	A	19771115	US 1974-457879	19740404
PRIORITY APPLN. INFO.:			US 1967-693148	A2 19671226
			US 1970-4179	A2 19700114
			US 1971-159515	A1 19710702

AB A gaseous olefin (e.g., **ethylene**, propene, and 2-butene) or acetylene was bubbled through liquid **perfluoroalkyl iodide** [e.g., 1-iodoperfluoroheptane (I) and 1-iodoperfluorohexane] or a mixture of **perfluoroalkyl iodides** at .apprx.50-100° in the presence of a free radical generating catalyst to give the corresponding adducts, essentially free of telomers. Thus, **ethylene** was bubbled through a mixture of I and Bz2O2 at 80-5° for .apprx.10.5 h to give 1,1,2,2-tetrahydro-1-iodoperfluorononane (88.5% yield) free of telomers. About 18 other fluoroiodoalkanes or -alkenes were similarly prepared

L7 ANSWER 22 OF 31 CAPLUS COPYRIGHT 2006 ACS on STN

ACCESSION NUMBER: 1974:48569 CAPLUS

DOCUMENT NUMBER: 80:48569

TITLE: Fluoroalkyl iodide telomers

INVENTOR(S): Rudolph, Werner; Massonne, Joachim

PATENT ASSIGNEE(S): Kali-Chemie A.-G.

SOURCE: Ger. Offen., 11 pp.

CODEN: GWXXBX

DOCUMENT TYPE: Patent

LANGUAGE: German

FAMILY ACC. NUM. COUNT: 1

## PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
DE 2218451	A1	19731031	DE 1972-2218451	19720417
CH 580552	A	19761015	CH 1973-4264	19730323
GB 1415245	A	19751126	GB 1973-16199	19730404
NL 7304737	A	19731019	NL 1973-4737	19730405
BE 798268	A1	19731016	BE 1973-130056	19730416
FR 2180863	A1	19731130	FR 1973-13754	19730416
JP 49014407	A2	19740207	JP 1973-43040	19730416
IT 982785	A	19741021	IT 1973-23107	19730417

PRIORITY APPLN. INFO.: DE 1972-2218451 A 19720417

AB Fluoroalkyl iodide telomers were prepared by 1:1 addition of **ethylene** to C6-12 **perfluoroalkyl iodide** telomers (I) in the presence of peracid catalysts, e.g. peracetic acid [79-21-0] at 70-100.deg.. Thus, 40 standard l. **ethylene**/hr and 30 g catalyst solution/hr (11.6% HO2Ac in C2F3Cl3) were passed into a reactor containing 4000 g I 8 hr at 70-80.deg. to give 98.3% monoethylene adduct of I.

L7 ANSWER 23 OF 31 CAPLUS COPYRIGHT 2006 ACS on STN

ACCESSION NUMBER: 1974:14545 CAPLUS

DOCUMENT NUMBER: 80:14545

TITLE: **Perfluoroalkyl iodides**

INVENTOR(S): Oda, Yoshio; Suhara, Manabu

PATENT ASSIGNEE(S): Asahi Glass Co., Ltd.

SOURCE: Jpn. Kokai Tokkyo Koho, 4 pp.

CODEN: JKXXAF

DOCUMENT TYPE:

Patent

LANGUAGE:

Japanese

FAMILY ACC. NUM. COUNT:

1

## PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
JP 48052706	A2	19730724	JP 1971-88200	19711108
JP 56002054	B4	19810117		

## PRIORITY APPLN. INFO.:

JP 1971-88200 A 19711108

AB Catalytic action of Mo or W fluoride gave **perfluoroalkyl iodides** from C<sub>2</sub>F<sub>4</sub>, iodine, and IF<sub>5</sub>. E.g., 20.3 g C<sub>2</sub>F<sub>4</sub> was fed to a mixture of iodine 16.8 g, IF<sub>5</sub> 7.4 g, and MoF<sub>6</sub> 100 mg at 75° in 2.8 hr at 4-12 kg/cm<sup>2</sup> to give 39.1 g pentafluoroethyl iodide.

L7 ANSWER 24 OF 31 CAPLUS COPYRIGHT 2006 ACS on STN

ACCESSION NUMBER: 1969:501287 CAPLUS

DOCUMENT NUMBER: 71:101287

TITLE: Iodoperfluoroalkanes

INVENTOR(S): Knell, Martin

PATENT ASSIGNEE(S): Geigy, J. R., A.-G.

SOURCE: Ger. Offen., 11 pp.

CODEN: GWXXBX

DOCUMENT TYPE: Patent

LANGUAGE: German

FAMILY ACC. NUM. COUNT: 3

## PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
DE 1816706	C3	19740926	DE 1968-1816706	19681223
CH 509954	A	19710715	CH 1968-509954	19681202
GB 1210730	A	19701028	GB 1968-1210730	19681224
FR 1597171	A	19700622	FR 1968-1597171	19681226

## PRIORITY APPLN. INFO.:

US 1967-693148 A 19671226

AB Perflouroalkyl iodides [C<sub>x</sub>F<sub>2x+1</sub>] (x = 4-14), which are liquid at reaction temperature, are added to gaseous olefins and acetylenes in the presence of a catalyst giving free radicals, at atmospheric or lower pressure and 50-220°. Thus, **ethylene** is introduced during 10.5 hrs. at 80-5°C. into a mixture of 10 parts by weight 1-iodoperfluoroheptane and 1 part by weight benzoyl peroxide to give 88.5% 1,1,2,2-tetrahydro-1-iodoperfluorononane b<sub>23</sub> 89-90°, m. 40-5°. Similarly prepared were: 1,1,1,2,3,3-hexahydro-2-iodoperfluorodecane, b. 119-20°; 1,1-dimethyl-2,2-dihydro-1-iodoperfluorononane, b. 82-4°; 1,2-dimethyl-1,2-dihydro-1-iodoperfluorononane, b. 86-90°; 1,1,2,2-tetrahydro-1-iodoperfluoroctane, b. 107-10°; 1,2-dihydro-1-iodoperfluorononene.

L7 ANSWER 25 OF 31 CAPLUS COPYRIGHT 2006 ACS on STN

ACCESSION NUMBER: 1969:402955 CAPLUS

DOCUMENT NUMBER: 71:2955

TITLE: Perfluoroalkyl fluoriodides and their use for telomerization of **perfluoroalkyl iodides** with olefins

INVENTOR(S): Rondestvedt, Christian S., Jr.

PATENT ASSIGNEE(S): du Pont de Nemours, E. I., and Co.

SOURCE: Fr., 14 pp.

CODEN: FRXXAK

DOCUMENT TYPE: Patent

LANGUAGE: French

FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
FR 1521775		19680419	FR 1967-104939	19670502

PRIORITY APPLN. INFO.:

AB The title preparation and telomerization, especially telomerization of tetrafluoroethylene (I) is described. Thus, 4.67 g. liquid ClF<sub>3</sub> was added with stirring at -70° to a solution of 33.4 g. n-C<sub>6</sub>F<sub>13</sub>I in 100 ml. perfluorohexane. The temperature raised by exothermic reaction to 0°, C<sub>6</sub>F<sub>14</sub> and excess C<sub>6</sub>F<sub>13</sub>I were evaporated in vacuo <0° to give C<sub>6</sub>F<sub>13</sub>IF<sub>2</sub>. Similarly, C<sub>2</sub>F<sub>5</sub>I and ClF<sub>3</sub> gave C<sub>2</sub>F<sub>5</sub>IF<sub>2</sub> with some C<sub>2</sub>F<sub>5</sub>IF<sub>4</sub>; 6 moles n-C<sub>4</sub>F<sub>9</sub>I with 1 mole ClF<sub>3</sub> gave n-C<sub>4</sub>F<sub>9</sub>IF<sub>2</sub>, m. 150°; 0.0337 mole n-C<sub>10</sub>F<sub>21</sub>I with 0.03 mole ClF<sub>3</sub> gave C<sub>10</sub>F<sub>21</sub>IF<sub>2</sub>, solid. Addition of 40.7 g. liquid ClF<sub>3</sub> to 206 g. n-C<sub>4</sub>F<sub>9</sub>I in 2614 g. C<sub>6</sub>F<sub>14</sub> at -80° gave a precipitate, dissolved by slow heating to -30°; the solution was cooled to -55°, and more 69.3 g. ClF<sub>3</sub> was added to give 80% n-C<sub>4</sub>F<sub>9</sub>IF<sub>4</sub> m. 10° (stable at -20°, but decomposed at room temperature). Dry He, saturated at 0° with ClF<sub>3</sub> vapor, was bubbled in C<sub>2</sub>F<sub>5</sub>I at 70° to absorb 2 moles ClF<sub>3</sub>, and the mixture was evaporated in vacuo <0° to give C<sub>2</sub>F<sub>5</sub>IF<sub>2</sub>, m. 18°. By the He-method, the reaction of 0.1 mole ClF<sub>3</sub> with 0.5 mole (CF<sub>3</sub>)<sub>2</sub>CFI at -70° gave 99% (CF<sub>3</sub>)<sub>2</sub>CFIF<sub>2</sub>, pasty solid; 0.1 mole ClF<sub>3</sub> with 0.5 mole C<sub>6</sub>F<sub>13</sub>I gave n-C<sub>6</sub>F<sub>13</sub>IF<sub>2</sub>, m. 35°; 25 g. CF<sub>3</sub>I in 30 ml. C<sub>6</sub>F<sub>14</sub> with He-ClF<sub>3</sub> at -80 to 30° gave a solid, decomposing 0°. A mixture of 42.6 g. C<sub>2</sub>F<sub>5</sub>I and 7 g. BrF<sub>5</sub> did not react at -50°; on heating, a violent reaction gave C<sub>2</sub>F<sub>5</sub>IF<sub>2</sub> and C<sub>2</sub>F<sub>5</sub>IF<sub>4</sub>. A similar reaction of 0.15 mole n-C<sub>4</sub>F<sub>9</sub>I with 0.06 mole BrF<sub>5</sub> at -80 to -30° gave an equimolar mixture of n-C<sub>4</sub>F<sub>9</sub>-IF<sub>2</sub> and C<sub>4</sub>F<sub>9</sub>IF<sub>4</sub>, m. 18°. The mixture of 0.1 mole n-C<sub>4</sub>F<sub>9</sub>I in 146 g. C<sub>6</sub>F<sub>14</sub> with 0.04 mole BrF<sub>5</sub> at -80 to 0° gave a mixture of 64% n-C<sub>4</sub>F<sub>9</sub>IF<sub>4</sub> and 36% n-C<sub>4</sub>F<sub>9</sub>IF<sub>2</sub>, m. 16°. A similar reaction of C<sub>4</sub>F<sub>9</sub>I with BrF<sub>3</sub> at the same conditions gave the same compds. For the telomerization, 5.9 g. C<sub>6</sub>F<sub>13</sub>IF<sub>2</sub> and 100 g. n-C<sub>3</sub>F<sub>7</sub>I in a glass autoclave in vacuo was heated to 70°, and 33.4 g. I was introduced at 70-8°/8.4 bar, in 10 min. After 32 min. at 70°, the pressure fell to 2.25 bar, to give a mixture of telomers F(CF<sub>2</sub>)<sub>n</sub>I with predominately odd n from 3 to 19. Other methods for telomerization of I are related, giving telomers with n 0-7, 1-6, 3-11, 2-20, with predominately odd or even n, using as telogen C<sub>4</sub>F<sub>9</sub>I and C<sub>2</sub>F<sub>5</sub>I, and as catalyst C<sub>4</sub>F<sub>9</sub>IF<sub>2</sub> and C<sub>3</sub>F<sub>7</sub>IF<sub>4</sub>. RIF<sub>2</sub> are more active as catalysts than RIF<sub>4</sub>. The telomerization of ethylene with n-C<sub>4</sub>F<sub>9</sub>I, catalyzed with n-C<sub>4</sub>F<sub>9</sub>IF<sub>2</sub>, at 60-80° gave a low yield of n-C<sub>4</sub>F<sub>9</sub>CH<sub>2</sub>CH<sub>2</sub>I and traces of telomers with n = 2,3,4. Similarly, vinyl fluoride gave n-C<sub>4</sub>F<sub>9</sub>CH<sub>2</sub>CH<sub>2</sub>IFI, and vinylidene fluoride gave n-C<sub>4</sub>F<sub>9</sub>CH<sub>2</sub>CF<sub>2</sub>I, b760 120°.

L7 ANSWER 26 OF 31 CAPLUS COPYRIGHT 2006 ACS on STN

ACCESSION NUMBER: 1966:43304 CAPLUS  
 DOCUMENT NUMBER: 64:43304  
 ORIGINAL REFERENCE NO.: 64:8031h,8032a-c  
 TITLE: **Perfluoroalkyl iodides**  
 INVENTOR(S): Blanchard, Wesley A.; Rhode, Judson C.  
 PATENT ASSIGNEE(S): E. I. du Pont de Nemours & Co.  
 SOURCE: 4 pp.  
 DOCUMENT TYPE: Patent  
 LANGUAGE: Unavailable  
 FAMILY ACC. NUM. COUNT: 1  
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
US 3226449		19651228	US 1962-200299	19620606

AB **Perfluoroalkyl iodides** containing 6-12 C atoms may be

prepared by injecting C<sub>2</sub>F<sub>4</sub> and a free radical-generating catalyst into a mixture of (1) a **perfluoroalkyl iodide** chosen from C<sub>2</sub>F<sub>5</sub>I and mixts. of C<sub>2</sub>F<sub>5</sub>I and C<sub>4</sub>F<sub>9</sub>I and (2) a portion of free radical generating catalyst. The mixture is heated to 80-170° under a pressure of 225-700 psi. The injection of the C<sub>2</sub>F<sub>4</sub> mixture is continued until there are 0.25-1.2 moles of C<sub>2</sub>F<sub>4</sub> per mole of **perfluoroalkyl iodide**. The amount of catalyst should be 0.25-0.9 weight-% of the total reaction mixture. Product yields of 70% or better are obtained. The preferred catalyst is di-tert-butyl peroxide. The preferred reaction temperature with the catalyst is 165°. The preferred mole ratio of C<sub>2</sub>F<sub>4</sub>-iodide is 0.4-1.1. The table gives yields obtained under various conditions. The products may be used as intermediates for preparing oil and water repellants for textiles and as intermediates for the preparation of perfluoroalkanecarboxylic acids which are surface active agents. Parts, 2F<sub>6</sub>I, C<sub>4</sub>F<sub>9</sub>I, Total tert-BuO<sub>2</sub> used, weight %, Temperature, Ratio of C<sub>2</sub>F<sub>4</sub> added to initial iodide moles, % yield C<sub>6</sub>F<sub>13</sub>I-C<sub>12</sub>F<sub>25</sub>I, Based on C<sub>2</sub>F<sub>4</sub>, Based on iodide; 800, 800, 0.9, 165°, 0.654, 93.9, 74.5; 1200, 400, 0.89, 165°, 0.961, 98, 80.1; 1200, 400, 0.78, 165°, 0.67, 98+, 73.6; 1200, 400, 0.85, 165°, 0.767, 90.7, 81.9; 1200, 400, 0.91, 165°, 0.86, --, 60; 1200, 400, 0.78, 175°, 1.2, 65, 65;

L7 ANSWER 27 OF 31 CAPLUS COPYRIGHT 2006 ACS on STN

ACCESSION NUMBER: 1966:18698 CAPLUS  
 DOCUMENT NUMBER: 64:18698  
 ORIGINAL REFERENCE NO.: 64:3349g-h,3350a-b  
 TITLE: **Perfluoroalkyl iodides**  
 INVENTOR(S): Bloechl, Walter  
 SOURCE: 13 pp.  
 DOCUMENT TYPE: Patent  
 LANGUAGE: Unavailable  
 FAMILY ACC. NUM. COUNT: 1  
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
NL 6414504		19650614	NL 1964-14504	19641211
PRIORITY APPLN. INFO.:			DE	19631212

AB The title compds. are prepared in high yields (without forming polymers) by treating a perfluoroolefin R<sub>1</sub>CF:CFR<sub>2</sub> with a saturated haloalkyl iodide I(CF<sub>2</sub>)<sub>n</sub>X or I(CFR<sub>3</sub>CFR<sub>4</sub>)<sub>m</sub>(CF<sub>2</sub>)<sub>n</sub>X at 260-350° for <10 min. Thus, a mixture of 2 volume parts gaseous perfluoroheptyl iodide (I) and 1 volume part CF<sub>2</sub>:CF<sub>2</sub> (II) was fed continuously (0.1 g. I/min.) into a Pyrex glass tube and heated at 260°. After a contact time of 3 min., the mixture was cooled to -10°. After 5 hrs., the liquid formed was distilled to yield 25.8 g. I, 4.05 g. perfluorononyl iodide (III), b<sub>760</sub> 178°, b<sub>40</sub> 85°, and b<sub>100</sub> 98°, and 1.1 g. residue, containing 90% perfluoroundecyl iodide (IV), b<sub>40</sub> 113°. At 390-410°, waxlike compds., m. >80° were formed. Similarly, CF<sub>3</sub>CF<sub>2</sub>I (V) and II (containing 0.3% O) were heated at 330°, and the product (300 g.) was distilled to yield 156 g. V, 51 g. 1-iodononafluorobutane, 41 g. 1-iodotridecafluorohexane, 23 g. 1-iodoheptadecafluoroctane, 15.5 g. F(CF<sub>2</sub>)<sub>10</sub>I, and 7 g. F-(CF<sub>2</sub>)<sub>12</sub>I. The perfluoro compds. prepared were (reactants, volume ratio of reactants, reaction conditions, yield, products formed, and phys. consts. are given): CF<sub>3</sub>I, II, 4:1, 40 sec./305°/4 atmospheric, --, perfluoroiodopropane (and some perfluoroiodopentane and a trace of I); I, perfluoropropene, 5:1 by weight, --/245°/300 atmospheric, --, 2-trifluoromethyl-1-iodononane, b<sub>120</sub> 129°; ICF<sub>2</sub>CF<sub>2</sub>H, II, 3:1, 3 min./260°/750 mm., 85% (13% conversion), H(CF<sub>2</sub>)<sub>4</sub>I, b<sub>750</sub> 85°; ICF<sub>2</sub>CF<sub>2</sub>Cl, II, 3:1, 3 min./260°/750 mm., 80% (9% conversion),

$\text{Cl}(\text{CF}_2)_4\text{I}$ , b350 76°; I( $\text{CF}_2$ )<sub>2</sub>I, II, 5:1, 3 min./260°/750 mm., --, I( $\text{CF}_2$ )<sub>4</sub>I, b100 86°. The compds. are useful as hydraulic fluids.

L7 ANSWER 28 OF 31 CAPLUS COPYRIGHT 2006 ACS on STN

ACCESSION NUMBER: 1965:90276 CAPLUS  
 DOCUMENT NUMBER: 62:90276  
 ORIGINAL REFERENCE NO.: 62:16051g-h,16052a  
 TITLE: **Perfluoroalkyl iodides**  
 PATENT ASSIGNEE(S): E. I. du Pont de Nemours & Co.  
 SOURCE: 22 pp.  
 DOCUMENT TYPE: Patent  
 LANGUAGE: Unavailable  
 FAMILY ACC. NUM. COUNT: 1  
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
FR 1380555	-----	19641204	FR 1963-949248	19631001
AB IF5 73.3 is mixed at room temperature with 167.7 I and 0.5 part $\text{SbF}_3$ in a bomb jacketed with Hastelloy C. The bomb is cooled at 0°, purged of air and heated at 80° with stirring. $\text{C}_2\text{F}_4$ (I) 192 parts is added portionwise over an hr. The pressure rises from 14 to 17 kg./cm. <sup>2</sup> after each addition. After another hr. the bomb is vented through a water cleaner followed by a drying column (Drierite). The gases are collected at -60°. The non-condensed gas contains 4.3 parts $\text{C}_2\text{F}_5\text{I}$ (II), while the condensed fraction (397 parts) contains 98.3% II, 0.4% I, 0.8% $\text{CF}_2\text{ICF}_2\text{I}$ and 0.4% $\text{C}_4\text{F}_{10}$ . The yield in II is 98.5% based on the consumed I. Other catalysts may be used (given: catalyst (2 parts) and yield in % II) $\text{SnF}_2$ , 99; $\text{CaF}_2$ , 78; $\text{CrF}_3\cdot 3\text{H}_2\text{O}$ , 16; $\text{KF}$ , 18; $\text{HF}$ (100 parts), 15; $\text{CoF}_2\cdot 2\text{H}_2\text{O}$ , 60; $\text{PbF}_4$ , 39; $\text{CuF}_2$ , 78; $\text{NiF}_2\cdot 5\text{H}_2\text{O}$ , 28; $\text{AgF}$ (50%), 73. Depending on the exptl. conditions, the following products were also obtained in the reaction mixture: $\text{C}_4\text{F}_9\text{I}$ , $\text{C}_6\text{F}_{13}\text{I}$ , $\text{C}_8\text{F}_{17}\text{I}$ , $\text{C}_{10}\text{F}_{21}\text{I}$ , $\text{C}_{12}\text{F}_{25}\text{I}$ , $\text{C}_{14}\text{F}_{29}\text{I}$ , $\text{C}_{16}\text{F}_{33}\text{I}$ .				

L7 ANSWER 29 OF 31 CAPLUS COPYRIGHT 2006 ACS on STN

ACCESSION NUMBER: 1964:410993 CAPLUS  
 DOCUMENT NUMBER: 61:10993  
 ORIGINAL REFERENCE NO.: 61:1755d-h,1756a-b  
 TITLE: Improvement in the preparation of **perfluoroalkyl iodides** from tetrafluoroethylene  
 INVENTOR(S): Parsons, Raymond E.  
 PATENT ASSIGNEE(S): E. I. du Pont de Nemours & Co.  
 SOURCE: 5 pp.  
 DOCUMENT TYPE: Patent  
 LANGUAGE: Unavailable  
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
US 3132185	-----	19640505	US 1962-191722	19620502
BE 640970	-----		BE	
GB 998235	-----		GB	

AB The preparation is given of perfluoroethyl iodide ( $\text{C}_2\text{F}_5\text{I}$ ) (I) and longer chain title compds.  $\text{C}_2\text{F}_5(\text{CF}_2\text{CF}_2)_n\text{I}$  (II), where n = 1 or greater, according to the reaction  $2\text{I}_2 + \text{IF}_5 + 5\text{CF}_2:\text{CF}_2 \rightarrow 5\text{CF}_3\text{CF}_2\text{I}$ . If the molar ratio of iodine to  $\text{IF}_5$  < 2:1, products of formula II (mixture, various n values) result, usually in conjunction with varying amts. of I depending on the iodine to  $\text{IF}_5$  ratio. Thus, 0.66 mole iodine, 0.33 mole  $\text{IF}_5$  (III), and 0.0028 mole  $\text{SbF}_5$  were mixed at ambient temperature in a Hastelloy-C lined autoclave (2 moles iodine and 8.48 millimoles catalyst/mole III), the

whole cooled <0°, evacuated to remove air, heated to 80° with agitation and CF<sub>2</sub>CF<sub>2</sub> (IV) and 192 parts added slowly; the pressure rose to 200-50 lb./in.<sup>2</sup> gage in <3 min., the temperature rose 8-26° after each addition in about 1 min. and then reverted to about 80° until 165 parts had been added. No temperature increase occurred during the addition of the

remaining 27 parts of IV. The mixture was kept 1 hr. at 80°, cooled and the volatile products collected in a refrigerated receiver at -60°. Mass-spectrometry, showed 98.3% I, 0.4% unreacted IV, 0.8% IF<sub>2</sub>CCF<sub>2</sub>I (V) and 0.4% perfluorocyclobutane (VI). The noncondensed gases contained 28.1 parts IV and 4.3 parts I. The yield of I was 98.5%, based on IV. When the reaction temperature was 60° rather than 80°, the condensed product contained 97.4% I, 0.7% IV, 0.6% VI, 0.7% V, and 0.4% other products; the yield of I was 98.1%. When the reaction was repeated with omission of the SbF<sub>5</sub>, the condensed product contained 98.5% I, 0.4% IV, 0.5% V and 0.5% VI, the oily residue contained 94.6% V and 4.4% I and the yield of I was 98.4% based on IV. The reaction was repeated with 0.0128 mole anhydrous SnF<sub>2</sub>; the condensed product contained 96.9% I, 0.6% IV, 0.8% VI, 0.3% V, and 1.3% other products. The yield of I was 99% based on IV. Similarly, other catalysts were used (catalyst, and amount (parts) and % yield of I given): CaF<sub>2</sub>, 2.0, 78; CrF<sub>3</sub>.3H<sub>2</sub>O, 2.0, 16; KF, 2.0, 18; HF, 100.0, 15; CoF<sub>2</sub>.2H<sub>2</sub>O, 2.0, 60; PbF<sub>4</sub>, 2.0, 39; CuF<sub>2</sub>, 2.0, 78; NiF<sub>2</sub>.5H<sub>2</sub>O, 2.0, 28; and AgF (50%), 2.0, 73. The following examples give the mixture of components. A mixture of 0.33 mole iodine, 0.182 mole III, and 0.0028 mole SbF<sub>3</sub> (VII) was prepared at ambient temperature in a Hastelloy-C lined autoclave,

cooled to below 0°, evacuated, sealed, heated to 60° with agitation, and 1.21 moles IV added slowly in small increments in 1 hr. The reaction was exothermic, the pressure dropped rapidly from 150-250 lb./in.<sup>2</sup> gage to <100 lb./in.<sup>2</sup> gage, the temperature rose 7-44° after each addition. The mixture was kept at 70° 1 hr., cooled, the volatile products were vented through a H<sub>2</sub>O scrubber and a CaSO<sub>4</sub> drying column, condensed in a refrigerated receiver at -60° and the condensed volatile material analyzed. It contained (in mole-%) 95.1 I, 1.2 C<sub>4</sub>F<sub>9</sub>I (VIII), 0.5 IV, and 1 C<sub>2</sub>F<sub>6</sub> (IX). The noncondensed gases were found to consist of 8.0 parts IV, 4.0 parts I, and 3.1 parts IX. The nonvolatile residue (100 parts) was washed with H<sub>2</sub>O and analyzed with a vapor phase chromatograph (in weight-%): I 21.1; VIII 28.7; C<sub>6</sub>F<sub>13</sub>I (X) 20.6; C<sub>8</sub>F<sub>17</sub>I (XI) 13.0; C<sub>10</sub>F<sub>21</sub>I (XII) 7.5; C<sub>12</sub>F<sub>25</sub>I (XIII) 4.1; C<sub>14</sub>F<sub>29</sub>I (XIV) 2.3; C<sub>16</sub>F<sub>33</sub>I (XV) 1.1. The combination of these analyses indicates that 96.9% IV is accounted for; % yields are as follows (based on IV consumed): I 48.8, VIII 15.8, X 12.3, XI 1.7, XV 0.9, and IX 0.6. Thus, the total yield of I was 95.8% and the total yield of products II was 47.1%. Iodine consumption was quant. Similarly, addnl. expts. were carried out with a varying ratio of iodine and III, as well as the temperature and the catalysts.

L7 ANSWER 30 OF 31 CAPLUS COPYRIGHT 2006 ACS on STN  
ACCESSION NUMBER: 1964:404316 CAPLUS  
DOCUMENT NUMBER: 61:4316  
ORIGINAL REFERENCE NO.: 61:684h,685a-b  
TITLE: Chemistry of the metal carbonyls. XXV. Fluorocarbon derivatives of nickel  
AUTHOR(S): McBride, D. W.; Dudek, E.; Stone, F. G. A.  
CORPORATE SOURCE: Harvard Univ.  
SOURCE: Journal of the Chemical Society (1964), (May), 1752-9  
CODEN: JCSOA9; ISSN: 0368-1769  
DOCUMENT TYPE: Journal  
LANGUAGE: Unavailable  
AB cf. CA 60, 5532e. π-C<sub>5</sub>H<sub>5</sub>.Ni(CO)Rf [Rf = CF<sub>3</sub>, C<sub>2</sub>F<sub>5</sub>, or n-C<sub>3</sub>F<sub>7</sub>] were isolated from reactions between dicarbonyl-π-dicyclopentadienyldinickel and **perfluoroalkyl iodides**. The related complexes were also obtained, namely, π-C<sub>5</sub>H<sub>5</sub>.Ni(CO)Me, π-C<sub>5</sub>H<sub>5</sub>.Ni(Ph<sub>3</sub>P)C<sub>2</sub>F<sub>5</sub>,

and  $\pi$ -C<sub>5</sub>H<sub>5</sub>.Ni(Ph<sub>3</sub>P)I. Treatment of the salt K[ $\pi$ -C<sub>5</sub>H<sub>5</sub>.Ni(CO)] with perfluoroallyl chloride gave a mixture of  $\pi$ -C<sub>5</sub>H<sub>5</sub>.Ni(CO)(CF<sub>2</sub>CF:CF<sub>2</sub>) and  $\pi$ -C<sub>5</sub>H<sub>5</sub>.Ni(CO)(CF:CFCF<sub>3</sub>). The probable constitution of the C<sub>4</sub>F<sub>5</sub> group in a new complex  $\pi$ -C<sub>5</sub>H<sub>5</sub>.Ni(Ph<sub>3</sub>P)C<sub>4</sub>F<sub>5</sub> was discussed. Reactions between dicyclopentadienylnickel and ethylene, hexafluorobut-2-yne, and butadiene were described.

L7 ANSWER 31 OF 31 CAPLUS COPYRIGHT 2006 ACS on STN

ACCESSION NUMBER: 1951:49720 CAPLUS

DOCUMENT NUMBER: 45:49720

ORIGINAL REFERENCE NO.: 45:8442a-f

TITLE: Synthesis of fluorocarbons, perfluoroalkyl iodides, bromides, and chlorides, and perfluoroalkyl Grignard reagents

AUTHOR(S): Haszeldine, R. N.

CORPORATE SOURCE: Univ. Cambridge, UK

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AB cf. C.A. 44, 3875h, 4861d. I(CF<sub>2</sub>CF<sub>2</sub>)<sub>2</sub>I is prepared in high yield from ICF<sub>2</sub>CF<sub>2</sub>I and C<sub>2</sub>F<sub>4</sub> with heat, also from C<sub>2</sub>F<sub>4</sub> and iodine by heating under pressure; in both cases higher members of the series I(CF<sub>2</sub>CF<sub>2</sub>)<sub>n</sub>I are formed in amts. that can be varied by choice of temperature and pressure. The cyclic dimer of C<sub>2</sub>F<sub>4</sub> is a by-product in these reactions. The reaction is more conveniently carried out in stages in which each member of the series is converted into the next higher member. With lower members of the series, small amts. of the compds. formed with 2 or more C<sub>2</sub>F<sub>4</sub> mols. are produced and this tendency becomes greater with longer chains. Compds. containing up to 9 C<sub>2</sub>F<sub>4</sub> units have been prepared. A chain mechanism is proposed for these reactions. Compds. of greater chain length, from 10 to 20 C<sub>2</sub>H<sub>4</sub> units, are obtained by heating iodine with excess C<sub>2</sub>F<sub>4</sub> than by heating C<sub>2</sub>F<sub>4</sub>I<sub>2</sub>, C<sub>4</sub>F<sub>8</sub>I<sub>2</sub>, etc., with excess C<sub>2</sub>F<sub>4</sub> or from C<sub>2</sub>F<sub>4</sub> and excess iodine. Compds. with the general formula I(CF<sub>2</sub>CF<sub>2</sub>)<sub>n</sub>I have properties similar to those of CF<sub>3</sub>(CH<sub>2</sub>)<sub>n</sub>I; the lower members are liquids and the higher members are waxy solids. The compds. with n greater than 1 are much more stable than C<sub>2</sub>F<sub>4</sub>I<sub>2</sub>, but the C-I bonds are broken by heat or ultraviolet light to give the corresponding free radicals which undergo addition-polymerization with C<sub>2</sub>H<sub>4</sub>, C<sub>2</sub>H<sub>2</sub>, and yield organometallic compds. I(CF<sub>2</sub>CF<sub>2</sub>)<sub>n</sub>I with IF<sub>5</sub>, SbF<sub>5</sub>, or F diluted with N yield CF<sub>3</sub>(CF<sub>2</sub>)<sub>n</sub>I. Fluorocarbons are conveniently prepared in high yield up to C<sub>18</sub>F<sub>38</sub> from I(CF<sub>2</sub>)<sub>n</sub>I or CF<sub>3</sub>(CF<sub>2</sub>)<sub>n</sub>I with F diluted with N, IF<sub>5</sub>, IF<sub>7</sub>, or SbF<sub>5</sub> in an autoclave or by passing over heated CoF<sub>3</sub>. Pos. iodine is present in perfluoroalkyl mono- and diiodides and it may be replaced with H from alc. alkali or by irradiation in solvents such as hydrocarbons, alcs., and ethers. Br(CF<sub>2</sub>CF<sub>2</sub>)<sub>n</sub>Br and Cl(CF<sub>2</sub>CF<sub>2</sub>)<sub>n</sub>Cl and small amts. of Br(CF<sub>2</sub>CF<sub>2</sub>)<sub>n</sub>I or Cl(CF<sub>2</sub>CF<sub>2</sub>)<sub>n</sub>I are formed by the thermal or photochem. bromination or chlorination. Perfluoroalkyl dibromides have been converted into perfluoroalkyl monobromides with BF<sub>3</sub>. Perfluoroalkyl monobromides give fluorocarbons with excess BF<sub>3</sub> or SbF<sub>5</sub> under pressure. The conversion of perfluoroalkyl dichlorides into perfluoroalkyl monochlorides and fluorocarbons with F diluted with N, ClF<sub>3</sub>, or SbF<sub>5</sub> under pressure gives poorer yields than are obtained from the corresponding iodo and Br compds. Oils, greases, and waxy solids are obtained from long-chain fluorocarbons with fluorochlorocarbons or with small amts. of cyclic fluorocarbons. The lower perfluoroalkyl monoiodides form Grignard reagents that behave normally with water, CO<sub>2</sub>, and acyl halides; the latter give low yields.

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